Journal of Soil Science and Plant Nutrition (2022) 22:3448–3462
https://doi.org/10.1007/s42729-022-00899-5

ORIGINAL PAPER

Long-Term Gypsum and Top-Dress Nitrogen Rates on Black Oat Forage Yield After Maize in No-Till

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Received: 9 August 2021 / Accepted: 2 June 2022 / Published online: 29 June 2022

Abstract

Nitrogen (N) fertilization is well studied for black oat (*Avena strigosa* Schreb.), but the crop response to gypsum and the combination of both is underexplored, especially in long-term studies under no-till. Topdressing N rates (0, 50, and 100 kg ha^{-1}) were applied for black oat over 2017 and 2018 crop seasons in a gypsum experiment (0, 3, 6, 9, and 12 Mg ha^{-1}) started in 2009. Soil chemical attributes, forage yield, macronutrients content, and apparent N recovery efficiency (RE) were evaluated. Gypsum increased Ca^{2+} and SO_4^{2-} in the soil and in the forage, while Mg^{2+} was decreased in the soil. N decreased in the soil but increased in the forage with gypsum, probably due to greater root growth and absorption potential, what also explains higher P and K in the forage. N-fertilization increased all macronutrients in the forage. The RE was higher with 50 kg$_{[N]}$ ha^{-1} than with 100 kg$_{[N]}$ ha^{-1}, while residual gypsum rates had minimal or no effect. Forage yield was higher with 3 Mg ha^{-1} (2017) and up to 6 Mg ha^{-1} (2018) of gypsum rates in the absense of N-fertilization. The long-term effect of 6 and 9 Mg ha^{-1} of gypsum presented higher yields with 50 and 100 kg$_{[N]}$ ha^{-1} which were mostly equivalent. Without gypsum, the yield was higher with 100 kg$_{[N]}$ ha^{-1}. Our hypothesis was not confirmed, and no synergistic effect was observed in forage yield and RE by black oat in response to the long-term effect of gypsum associated with N-fertilization.

Keywords Phosphogypsum · Urea · Haylage · No-tillage system

1 Introduction

To meet the global demand for food, crop yields must be increased while the ability of soils to produce needs to be kept in the long term (Kopittke et al. 2019). This can be achieved through the improvement in management practices, and no-till (NT) system is a good example. Black oat is widely cultivated under NT in southern Brazil, due its high adaptability to subtropical winter conditions, fast growth, high production of biomass, forage aptitude, and residue for soil mulch. Nitrogen (N) fertilization has been studied to improve black oat yield, while gypsum ($CaSO_4.2H_2O$) has been studied to improve soil fertility under NT (Vicensi et al. 2020b), but the interaction between these practices has been underexplored, especially in long-term studies.

Black oat can be used as cover crop for NT or for cattle grazing and preserved feed production. In any case, its residual biomass may release important amounts of N, P, and K (Ferreira et al. 2014) already at 30 days from harvest or burndown, what benefits nutrient cycling for the succeeding crop. The residues deposited on soil surface contribute for soil cover, a key feature of NT, together with crop rotation and minimum tillage (Ward et al. 2018).

The NT system is used on more than 33 million hectares in Brazil, and whereas it reduces erosion and increases soil organic matter (IBGE 2021), in the absence of tillage lime, fertilizers and crop residues stay on topsoil layer, so

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nutrients concentrate close to surface, while deeper layers get more acidic and less fertile (Zoca and Penn 2017).

In subtropical areas under NT, gypsum has been used in a complementary way to liming (Tiecher et al. 2018). It has relatively good solubility (2.5 g L^{-1}), which is about 200 times higher compared to limestone (Zoca and Penn 2017). It adds calcium (Ca^{2+}) and sulfur (S-SO_4^{2-}), reduces aluminum (Al^{3+}) activity, mobilizes magnesium (Mg^{2+}), and may mobilize potassium (K^+) from surface to deeper soil layers (Crusciol et al. 2016; Dalla Nora et al. 2017; Zoca and Penn 2017), improving the basic cations availability in the profile (Vicensi et al. 2020a; Fontoura et al. 2019). Gypsum has a positive impact on soil fertility that can increase root growth and crop performance (Vicensi et al. 2016), but the long-term aspects of gypsum use need to be better understood, especially in the ferralsols (Oxisols) under NT.

The soil diagnostic attributes for gypsum recommendation in Brazil has historically been high levels of Al^{3+} and/or low levels of Ca^{2+} in subsurface diagnostic soil layer (e.g., 0.2–0.4 m). However, even in the absence of these conditions, the application of gypsum in NT Oxisols has been reported to improve crop yield of Poaceae species such as wheat (*Triticum aestivum* L.), maize (*Zea mays* L.), barley (*Hordeum vulgare* L.) and black oats (*Avena strigosa* Schreb.), especially in crop seasons under water restriction conditions (Vicensi et al. 2016).

Poaceae species get most of their nitrogen (N) through N-fertilization. Being the most absorbed nutrient by many crops, N commonly limits plant production (Taiz et al. 2017), but many farmers apply lower N amounts than required for optimal production. Black oat, particularly, is often managed with low inputs in subtropical Brazil and grows under the residual effect of predecessor crop fertilization. Succeeding soybean, black oat has been reported to respond to N-fertilization in terms of nutrient uptake, biomass yield, root growth (Vicensi et al. 2020a, b, 2021b), nutritional status (Vicensi et al. 2021a), and nutrient cycling (Vicensi et al. 2021b). As Poaceae species like maize have crop residues with high carbon:nitrogen (C:N) ratio, the decomposition process causes N immobilization (Sprunger et al. 2019), what may increase succeeding black oat response to N-fertilization.

The yield of black oat aboveground biomass has been reported to increase with N rates (Vicensi et al. 2021b) in subtropical areas of southern Brazil, depending upon environmental conditions and production system. As fertilizer costs are expensive, and the negative effects of N loss processes are well known, especially when higher N rates are used (Huddell et al. 2020), the search for greater efficiency in the use of N has been sought, so that more environmentally friendly N rates are used, while economic viability is kept, and yield levels are enhanced.

As N is easily leached into the soil profile in the form of nitrate (NO_3^-) under normal aeration conditions (Wang et al. 2019) and considering that gypsum increases root growth of plants such as maize (Caires et al. 2016) and black oat (Vicensi et al. 2020b), we hypothesized that the association of the long-term effect of gypsum with topdressing N rates could have a synergistic effect in terms of forage yield and apparent N recovery by black oat. The objectives of this study were to evaluate topdressing N rates on black oat under no-till, inside a long-term gypsum experiment, assessing (i) soil fertility, (ii) forage yield, (iii) nutrient content, and (iv) apparent N recovery, in a field previously cultivated with maize.

2 Materials and Methods

2.1 Site Description

The data was collected based on a long-term study established in 2009 in Guarapuava, Paraná State, Brazil (25°23′S, 51°30′W, altitude 1026 m), in a humid subtropical mesothermic climate (Cfb-Köppen). The area was cultivated under rainfed conditions, and until the 2017 and 2018 crop seasons, considered for this study, the field was under continuous NT for more than 13 years, which are common agricultural conditions in the region.

The soil at the experiment site was classified as a very clayey Typic Hapludox (USDA Soil Survey), as well as Haplic Ferralsol (World Research Database—International Union of Soil Science). Results of soil morphological, chemical, and granulometric characterization up to 1.4 m (Michalovicz et al. 2019) presented that the profile had no physical limitations, had low Al^{3+} levels, and had low acidity in the A horizon due to anthropic activities. The diagnostic layer for annual crops (0–0.2 m) was sampled and analyzed in November 2009 (Table 1), in order to characterize the initial soil chemical conditions, and the soil test results for the 2015 and 2016 crop seasons (Vicensi et al. 2020a, 2020b) were used to guide the black oat fertilization in 2017 and 2018, as annual winter grass forage (SBCS/NRS 2016).

2.2 Experimental Design and Treatments

A complete randomized block design was used, in a split-plot arrangement with four replications. The plots (10 m × 6.4 m)

Table 1 Chemical characterization of the diagnostic layer (0–0.2 m) of the Typic Hapludox at the experimental site in Guarapuava, Paraná State, Brazil, 2009

Depth	P (Mehlich-1)	O.M	pH	Al	H + Al	Ca	Mg	K	CEC	SBS	Clay
m	mg dm^{-3}	g dm^{-3}	$CaCl_2$	cmolc dm^{-3}						%	g kg^{-1}
0–0.1	20.7	49.8	5.3	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	5.1	4.5	2.4	0.3	12.3	58.3	720

OM, organic matter; *CEC*, cation exchange capacity at pH 7.0; *SBS*, soil base saturation consisted of gypsum rates: 0, 3, 6, 9, and 12 Mg ha^{-1} of dry weight (G_0, G_3, G_6, G_9, and G_{12}, respectively), broadcasted on the soil surface in November 2009, November 2010, and November 2011, one-third rate each year after sowing summer crops. The rates represented 0, 33, 66, 100, and 133% of Ca^{2+} amount required to reach 60% of Ca saturation on the cation exchange capacity (CEC, pH 7.0) in the A_1 horizon. The subplots (3.33 m × 6.40 m) consisted of nitrogen (N) topdress rates 0, 50, and 100 $kg_{[N]}$ ha^{-1} ($N_{0,}$ N_{50}, and N_{100}, respectively), equivalent to 0%, 50%, and 100% of the N rate recommended for black oats at the beginning of the tillering stage (SBCS/NRS 2016). The N source was urea [($CO(NH_2)_2$), 45% of N].

2.3 Black Oat Sowing

The black oat (cultivar IAPAR 61) was sown on June 20 and July 12 in the 2017 and 2018 crop seasons, respectively, succeeding maize in both seasons. It was used a seeding rate of 360 seeds m^{-2} and a row spacing of 0.17 m. Furrow fertilization consisted of 40 kg ha^{-1} of P_2O_5 (triple superphosphate) banded with the seed. Before crop emergence, it was broadcasted 40 kg ha^{-1} of K_2O (potassium chloride) on the soil surface.

2.4 Soil Chemical Attributes

The soil was sampled between 7 and 10 days after N fertilization, at six points in the useful area of each subplot, with the following stratification: 0.0–0.1 and 0.1–0.2 m, using a Gouge auger; 0.2–0.4; 0.4–0.6, and 0.6–0.8 m using a Dutch auger. The composite samples were dried in a forced-air oven at 45 °C, grounded in a knife-type mill and sieved (2 mm mesh). Exchangeable Ca^{2+} and Mg^{2+} were extracted by 1 mol L^{-1} KCl and determined by atomic absorption spectroscopy. The $S\text{-}SO_4{}^{2-}$ fraction was extracted by 0.01 mol L^{-1} $Ca_3(PO_4)_2$ and determined by turbidimetry. Mineral-N ($NO_3{}^{-} + NH_4{}^{+}$) was extracted by 1 mol L^{-1} KCl and determined by Kjeldahl method.

2.5 Forage Yield

The early heading period, when winter forages are cut to produce haylage, was determined as stage 10.1 on the Feekes-Large Scale (Large 1954). Black oat reached this condition at 115 and 120 days after emergence in 2017 and 2018 crop seasons. Plants were cut 0.1 m from the soil surface in three points of 0.17 m^2 in the useful area of each subplot. Samples were pooled and dried in a forced air oven at 50 °C until constant weight was achieved, and the values were used to estimate dry matter yield of forage.

Aliquots of the dry matter samples were ground in a Wiley mill, sieved (0.75 mm), and analyzed for nitrogen (N) after sulfuric acid digestion (digestion block) and for phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), and sulfur (S) after nitric digestion (microwave) using standard methods. Nutrient concentrations were combined with forage yield values to determine the plant nutrient contents.

The apparent nitrogen recovery efficiency (RE) was calculated based on the forage yield response to N fertilization, as proposed by Congreves et al. (2021) according to the following equation:

$$\mathrm{RE}(\mathrm{kg\ kg^{-1}}) = \left[\frac{(\text{N accumulated in fertilized treatments}) - (\text{N accumulated in control})}{\text{Amount of N applied as fertilizer}}\right]$$

2.6 Meteorological Conditions

Rainfall (Fig. 1a, b), temperature (Fig. 1c, d), and solar radiation (Fig. 1e, f) data were obtained from a meteorological station (*SIMEPAR*/Brazil), located 200 m far from the experiment. A sequential water balance (Thornthwaite and Matter 1955) was calculated to identify water deficit periods during the crop growth cycles (Fig. 1a, b).

A total of 450.6 mm and 414.2 mm rainfall was recorded in the 2017 and 2018 crop seasons, respectively (Fig. 1a, b). For black oat, the evaporative demand in a 120-day growth cycle is around 450 mm (Bacchi et al. 1996), slightly unmet in the 2018 crop season. Actual evapotranspiration was below potential evapotranspiration in both crop seasons, especially in the 2017 crop season, with an accumulated water deficit of − 433.8 mm and − 145.3 mm in the 2017 and 2018 crop seasons, respectively (Fig. 1a, b). The mean average temperature in the 2017 and 2018 crop seasons was 16.2 °C and 16.3 °C, respectively (Fig. 1c, d). Considering a basal temperature of 5 °C for black oats (Peltonen-Sainio 1999), the accumulated degree days from the emergence until cutting was 1457.6 °C and 1305.3 °C in the 2017 and 2018 crop seasons, respectively. The accumulated solar radiation was 1815 MJ m^{-2} and 1677 MJ m^{-2} in the 2017 and 2018 crop seasons, respectively (Fig. 1e, f).

The conditions of temperature (Fig. 1c, d) were favorable for the development of black oats in most of the growth cycle. Water deficit occurred in both crop seasons, but more severely in the 2017 (Fig. 1a, b).

2.7 Statistical Analysis

The data were submitted to the Shapiro–Wilk test ($p \leq 0.05$) to verify the normality and homoscedasticity of the variance, and when they did not meet this condition, they were transformed according to Box-Cox test and then subjected to analysis of variance (ANOVA). For soil chemical attributes, ANOVA was carried out considering depth as a fixed factor. When means differed significantly at $p \leq 0.05$, N treatments

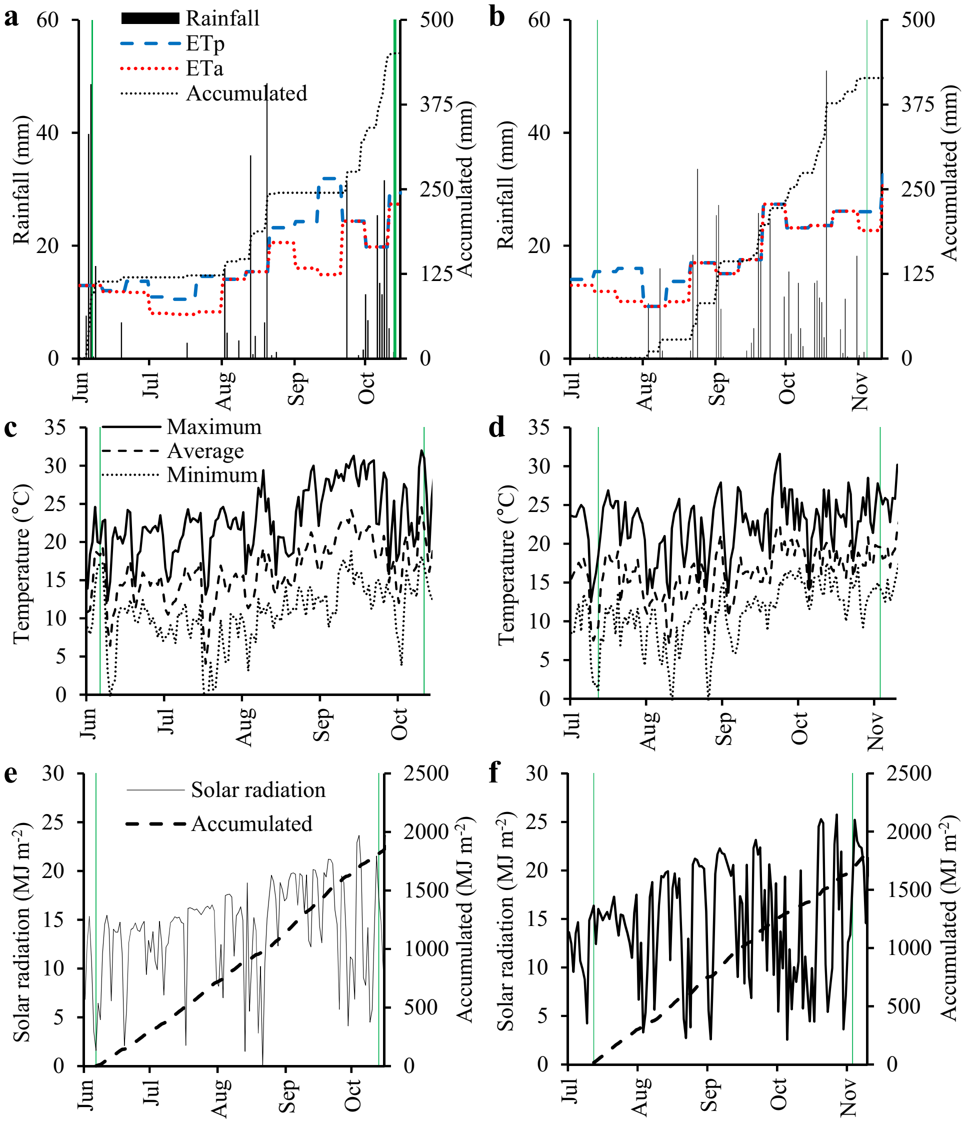

Fig. 1 Meteorological data of rainfall, accumulated rainfall (black dotted line), potential evapotranspiration (ETp, blue line), real evapotranspiration (ETa, red line); daily maximum (continuous line), average and minimum (dotted, respectively) temperatures; daily total solar radiation (continuous line) and accumulated solar radiation (dotted line) across the 2017 (**a**, **c**, **e**) and 2018 (**b**, **d**, **f**) crop seasons. Green lines indicate black oat sowing and cutting dates

were compared using the Tukey test ($\alpha = 0.05$), meanwhile gypsum treatments were fitted to linear or quadratic regression models, based on significance ($p \leq 0.05$ by the F test) and coefficient of determination (R^2).

3 Results

3.1 Soil Chemical Properties

The levels of Ca^{2+}, Mg^{2+}, and $S\text{-}SO_4^{-2}$ in the soil were not affected by N-fertilization (Table 2; Fig. S1,S2). The Ca^{2+} levels, only in 2017 crop season, increased in response to increasing long-term gypsum rates in all evaluated soil layers (Tables 2 and 3). The Mg^{2+} levels reduced with the increase in gypsum rates, in surface and subsurface soil layers in both evaluated crop seasons (Tables 3 and 4). The $S\text{-}SO_4^{-2}$ levels increased with the increase in gypsum rates in both evaluated crop seasons (Tables 3 and 4), with a cumulative effect in deeper layers (0.2–0.8 m).

The mineral-N levels presented interaction between long-term gypsum and N-fertilization in all evaluated soil layers and in both crop seasons (Table 2; Fig. S3). In general, in the surface layer (0–0.1 m) the effect of nitrogen fertilization o mineral-N levels was predictable, and it was higher at the rates of N_{100} and N_{50} compared to control (N_0) in all gypsum rates and in both crop seasons. In 2017 crop season, the mineral-N interaction within gypsum rates was as follows in the 0.0–0.1 m layer: in $G_{0,}$ G_9, and G_{12} $N_{100} = N_{50} > N_{0;}$ in G_3 and G_6 $N_{50} > N_{100} > N_0$ (Table 5). As for the interaction within N-fertilization was as follows, within N_0 and N_{50}, a quadratic and a linear model fitted best the gypsum rate, respectively, whereas N_{100} had no model adjustment (Table 5). In 2018

Table 2 Significance (*p* values) from analysis of variance of calcium (Ca^{2+}), magnesium (Mg^{2+}), sulfate ($S\text{-}SO_4^{2-}$), and mineral-N ($NH_4^+ + NO_3^-$) soil levels in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates top-dressed at tillering, under no-tillage Typic Hapludox during the 2017 and 2018 crop seasons in Guarapuava, Paraná state, Brazil

Treatment	Crop season	Soil chemical properties			
		Ca^{2+}	Mg^{2+}	$S\text{-}SO_4^{2-}$	mineral-N
Gypsum (G)	2017	**0.013***	**<.0001*****	**<.0001*****	**0.002****
	2018	0.245^{ns}	**0.019***	**0.002*****	**<.0001*****
Nitrogen (N)	2017	0.429^{ns}	0.579^{ns}	0.487^{ns}	**<.0001*****
	2018	0.159^{ns}	0.440^{ns}	0.923^{ns}	**<.0001*****
Soil depth (D)	2017	**<.0001*****	**<.0001*****	**<.0001*****	**<.0001*****
	2018	**<.0001*****	**<.0001*****	**<.0001*****	**<.0001*****
G×N	2017	0.553^{ns}	0.306^{ns}	0.895^{ns}	**<.0001*****
	2018	0.736^{ns}	0.644^{ns}	0.996^{ns}	**<.0001*****
G×D	2017	0.086^{ns}	**0.001****	**<.0001*****	**<.0001*****
	2018	**0.040***	**0.002****	**0.004****	**<.0001*****
N×D	2017	0.519^{ns}	0.926^{ns}	0.091^{ns}	**<.0001*****
	2018	**0.030***	**0.033***	0.995^{ns}	**<.0001*****
G×N×D	2017	0.534^{ns}	0.754^{ns}	0.888^{ns}	**<.0001*****
	2018	0.107^{ns}	0.290^{ns}	0.999^{ns}	**<.0001*****
CV (%)	2017	22	20	22	8
	2018	21	33	24	11

Bold numbers are statistically significant. ***, **, * and ^{ns} denote significant values at $p \leq 0.001$, $p \leq 0.01$, $p \leq 0.05$, and non-significant values, respectively

crop season, in the 0.0–0.1 m layer, the mineral-N interaction within gypsum rates was as follows: in G_0, G_3, G_6, and G_9 $N_{100} > N_{50} = N_0$ and in G_{12} $N_{100} = N_{50} > N_0$ (Table 5). As for the interaction within both N-fertilization, a quadratic model fitted best the gypsum rates (Table 5).

In the subsurface layers (0.1–0.8 m), the effect of N fertilization on mineral-N was not consistent, and in some cases, the mineral-N levels in control (N_0) were equal to or greater than the fertilized treatments (N_{50} and N_{100}) due to the depletion effect of long-term application of gypsum (Tables 2 and 5).

3.2 Black Oat Forage Yield

Black oat forage yield presented interaction between long-term applied gypsum and N-fertilization in both crop seasons (Fig. 2a, b). In 2017 crop season, the interaction effect on forage yield within gypsum rates was as follows: within G_0 $N_{100} \geq N_{50} \geq N_0$; within G_3 $N_{100} = N_{50} = N_0$; within both G_6 and G_9 $N_{100} = N_{50} > N_0$; and within G_{12} $N_{50} \geq N_{100} \geq N_0$ (Fig. 2a). Meanwhile, the interaction within N rates was as follows: no model was adjusted for N_0, whereas quadratic models were adjusted for gypsum rates in N_{50} and N_{100} (Fig. 2a).

In 2018 crop season, the interaction effect within gypsum rates was as follows: in G_0 and G_9 $N_{100} > N_{50} > N_0$ and in G_3, G_6, and G_{12} $N_{100} = N_{50} > N_0$ (Fig. 2b). Meanwhile, the interaction within N rates was as follows: quadratic models fitted best the gypsum rates in N_0 and N_{50}, and no model was adjusted for N_{100} (Fig. 2b). The models adjusted for gypsum rates presented low power of explanation (R^2), even considering significant effects of interaction in both crop seasons.

3.3 Forage Nutrient Content

The N, K, and Ca content were affected by long-term gypsum, whereas N-fertilization affected N, K, Ca, and Mg contents (Table 6). N and K contents did not present linear nor quadratic adjustment between the gypsum rates in both crop seasons. N content between N-fertilization rates were $N_{50} = N_{100} > N_0$ and $N_{100} > N_{50} > N_0$ in 2017 and 2018 crop seasons, respectively (Table 6). K content between N-fertilization rates were $N_{100} = N_{50} = N_0$ and $N_{100} > N_{50} > N_0$ in 2017 and 2018 crop seasons, respectively (Table 6). Ca and Mg contents fitted best in linear models to the gypsum rate, whereas between N-fertilization rates were $N_{100} = N_{50} > N_0$ in both crop seasons (Table 6).

P and S content presented interaction between long-term applied gypsum and N-fertilization (Table 7). In 2017 crop season, within gypsum rates P content were in G_0 $N_{100} \geq N_0 \geq N_{50}$; in G_3 $N_{100} = N_{50} = N_0$; in both G_6 and G_9 $N_{50} \geq N_0 \geq N_{100}$; and in G_{12} $N_{50} = N_{100} > N_0$ (Table 7). Within N fertilization rates, in N_{50} and N_{100} in both in a linear and quadratic model fitted best the gypsum rates, whereas N_0 presented no adjustment (Table 7). In 2018, the effect within gypsum rates for P content was in G_0 and G_9 $N_{100} > N_{50} = N_0$; in G_3 $N_{100} = N_{50} = N_0$; in G_6 $N_{50} = N_{100} > N_0$; and in G_{12} $N_{50} > N_{100} > N_0$ (Table 7). Within N rates, in N_0 a quadratic model and in N_{50} and N_{100} a linear model fitted best the gypsum rates (Table 7).

Table 3 Levels of Ca^{2+}, Mg^{2+}, and $S\text{-}SO_4^{2-}$ at 2017 crop season in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates top-dressed at tillering (N_0, N_{50}, and N_{100} refers to 0, 50, and 100 kg ha^{-1} of N) in a no-till Typic Hapludox in Guarapuava, Paraná state, Brazil

Depth (m)	Gypsum (Mg ha^{-1})	Ca^{2+} (cmol$_c$ dm^{-3})			Mg^{2+} (cmol$_c$ dm^{-3})			$S\text{-}SO_4^{2-}$ (mg dm^{-3})		
		N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}
0–0.1	0	3.1	2.4	1.9	1.0	0.8	0.5	13	14	15
	3	3.3	2.9	2.4	0.7	0.8	0.6	16	17	17
	6	2.6	2.2	2.5	0.6	0.5	0.5	20	20	19
	9	3.3	3.3	3.3	0.6	0.7	0.7	16	16	18
	12	3.5	3.9	3.2	0.6	0.6	0.6	18	17	15
Linear (R^2)		0.16^{**}			0.05^{ns}			0.07^{*}		
Quadratic (R^2)		0.17^{*}			0.09^{ns}			0.26^{*}		
0.1–0.2	0	3.1	2.4	2.6	1.0	0.8	0.6	17	17	18
	3	3.2	4.0	3.3	0.8	0.9	0.8	17	16	19
	6	2.4	1.7	3.3	0.6	0.5	0.6	24	24	24
	9	3.4	3.5	3.8	0.7	0.5	0.7	15	17	22
	12	4.1	4.4	3.4	0.4	0.6	0.4	19	26	24
Linear (R^2)		0.12^{**}			0.19^{***}			0.15^{**}		
Quadratic (R^2)		0.13^{*}			0.20^{**}			0.16^{**}		
0.2–0.4	0	2.3	2.2	1.9	0.9	0.8	0.6	27	29	29
	3	2.8	2.7	2.4	0.8	0.8	0.7	26	27	31
	6	2.2	2.2	2.6	0.4	0.5	0.6	41	45	40
	9	2.7	3.3	2.9	0.5	0.6	0.5	32	32	32
	12	3.3	3.2	3.0	0.4	0.5	0.4	39	39	38
Linear (R^2)		0.13^{**}			0.22^{***}			0.14^{**}		
Quadratic (R^2)		0.13^{*}			0.22^{**}			0.16^{**}		
0.4–0.6	0	1.6	1.5	1.6	0.9	0.6	0.7	42	44	44
	3	2.4	2.2	1.8	0.9	0.9	0.7	45	47	46
	6	2.1	2.1	2.1	0.4	0.4	0.5	65	67	61
	9	2.5	2.5	2.1	0.4	0.4	0.5	61	63	63
	12	3.1	3.0	2.5	0.4	0.5	0.3	62	69	69
Linear (R^2)		0.44^{***}			0.24^{***}			0.34^{***}		
Quadratic (R^2)		0.44^{**}			0.24^{**}			0.36^{***}		
0.6–0.8	0	1.3	1.4	1.5	0.9	0.8	0.8	45	47	43
	3	2.0	1.8	1.7	1.0	1.0	0.8	69	55	67
	6	2.0	2.1	2.1	0.6	0.6	0.6	79	79	81
	9	2.4	2.9	2.3	0.6	0.6	0.7	90	76	83
	12	2.4	2.7	2.5	0.5	0.5	0.4	77	63	91
Linear (R^2)		0.50^{***}			0.36^{***}			0.40^{***}		
Quadratic (R^2)		0.52^{**}			0.36^{**}			0.56^{***}		

Lowercase letters compare the N-fertilization averages within each crop season; ^{ns}, not significant. ***, **, * and ns denote significant values at $p \leq 0.001$, $p \leq 0.01$, $p \leq 0.05$, and non-significant values, respectively. Additional information on the Ca and Mg levels are presented in Fig. S1, whereas on S levels in Fig. S2

In 2017 crop season, within gypsum rates S content were in G_0, G_3, and G_6 $N_{100} = N_{50} = N_0$; in G_9 $N_{100} > N_{50} = N_0$; and in G_{12} $N_{100} > N_{50} > N_0$ (Table 7). Within N fertilization rates, in N_{50} and N_{100}, a linear model fitted best the gypsum rate, whereas N_0 presented no adjustment (Table 7). In 2018 crop season, within gypsum rates S content were in G_0 $N_{100} > N_{50} = N_0$; in G_3 and G_6 $N_{100} = N_{50} = N_0$; in G_9 $N_{100} \geq N_{50} \geq N_0$; and in G_{12} $N_{100} > N_{50} > N_0$ (Table 7). Within N fertilization rates, a quadratic model fitted best the gypsum rate for N_0, N_{50}, and N_{100} (Table 7).

3.4 Apparent N Recovery Efficiency

Apparent N recovery efficiency (RE) presented interaction between long-term gypsum and N-fertilization in both crop seasons (Table 8). Within gypsum rates, RE were over all gypsum rates as $N_{50} > N_{100}$ in 2017; and in 2018, RE were in G_0, G_3, and G_6 $N_{50} > N_{100}$; G_9 and G_{12} $N_{50} = N_{100}$ (Table 8). Within N fertilization rates in 2017 crop season, no models were adjusted for N_{50} and N_{100}. In 2018 crop season, within

Table 4 Levels of Ca^{2+}, Mg^{2+} and $S\text{-}SO_4^{2-}$ at 2018 crop season in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates top-dressed at tillering (N_0, N_{50}, and N_{100} refers to 0, 50, and 100 kg ha^{-1} of N) in a no-till Typic Hapludox in Guarapuava, Paraná state, Brazil

Depth (m)	Gypsum (Mg ha^{-1})	Ca^{2+} (cmol$_c$ dm^{-3})			Mg^{2+} (cmol$_c$ dm^{-3})			$S\text{-}SO_4^{2-}$ (mg dm^{-3})		
		N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}
0–0.1	0	2.1	1.8	3.0	0.8	0.6	0.9	14	13	12
	3	2.1	3.3	3.3	0.5	0.9	0.9	15	16	17
	6	2.5	2.0	2.3	0.4	0.4	0.5	17	20	20
	9	2.4	3.0	3.4	0.4	0.5	0.6	14	16	16
	12	2.0	3.0	3.3	0.5	0.7	0.7	14	16	14
Linear (R^2)		ns			0.08^{ns}			0.01^{ns}		
Quadratic (R^2)		ns			0.16^{ns}			0.23^{*}		
0.1–0.2	0	2.4	2.0	3.1	0.6	0.5	1.0	16	18	14
	3	3.5	3.9	3.3	0.8	1.0	0.8	13	14	13
	6	3.3	2.7	2.3	0.8	0.3	0.4	22	19	19
	9	3.1	3.3	3.5	1.0	0.5	0.5	14	15	17
	12	2.8	3.6	3.9	0.4	0.6	0.6	19	17	18
Linear (R^2)		ns			0.04^{ns}			0.07^{*}		
Quadratic (R^2)		ns			0.04^{ns}			0.07^{*}		
0.2–0.4	0	1.9	2.4	2.0	0.7	0.7	0.7	21	26	27
	3	2.4	2.6	2.1	0.6	0.8	0.8	18	20	30
	6	2.4	2.7	2.4	0.6	0.4	0.4	39	44	43
	9	2.6	3.0	3.2	0.4	0.4	0.4	32	31	35
	12	2.4	2.2	2.4	0.4	0.3	0.3	36	38	41
Linear (R^2)		ns			0.30^{***}			0.16^{**}		
Quadratic (R^2)		ns			0.30^{**}			0.17^{*}		
0.4–0.6	0	2.2	1.9	1.8	0.7	0.8	0.8	39	38	42
	3	2.2	2.0	2.1	0.7	0.8	0.8	46	41	48
	6	2.0	2.1	2.3	0.6	0.5	0.4	61	65	59
	9	2.1	2.7	2.7	0.4	0.4	0.4	76	68	81
	12	2.1	2.5	2.4	0.3	0.4	0.4	69	73	66
Linear (R^2)		ns			0.38^{***}			0.23^{***}		
Quadratic (R^2)		ns			0.39^{**}			0.24^{***}		
0.6–0.8	0	1.7	1.6	1.6	0.9	0.9	0.9	40	50	44
	3	2.1	1.8	2.4	0.9	1.0	1.2	52	51	47
	6	2.1	2.1	2.2	0.6	0.6	0.5	61	57	54
	9	2.5	2.6	2.6	0.5	0.6	0.5	78	76	72
	12	1.9	2.1	2.0	0.4	0.3	0.5	86	89	85
Linear (R^2)		ns			0.50^{***}			0.22^{***}		
Quadratic (R^2)		ns			0.50^{**}			0.23^{***}		

Lowercase letters compare the N-fertilization averages within each crop season; ^{ns}, not significant. ***, **, * and ns denote significant values at $p \leq 0.001$, $p \leq 0.01$, $p \leq 0.05$, and non-significant values, respectively. Additional information on the Ca and Mg levels are presented in Fig. S1, whereas on S levels in Fig. S2

N_{50}, a quadratic model fitted best the gypsum rates, whereas N_{100} presented no adjustment (Table 8).

4 Discussion

The increase in Ca^{2+} levels as a result of gypsum ($CaSO_4.2H_2O$) application has been widely reported for tropical and subtropical soils, especially in subsurface layers (Crusciol et al. 2016; Fontoura et al. 2019; Michalovicz et al. 2019), and was also observed by Vicensi et al. (2020a) previously in this experiment, indicating long-term effects of gypsum application. Cations like Ca^{2+} and Mg^{2+} react with SO_4^{2-} to form ionic pairs with zero net charge, which are more easily leached in the soil by water infiltration (Zoca and Penn 2017), with cumulative effects along time.

Ca^{2+} levels indicate further Ca^{2+} mobilization in depth from the 2017 to the 2018 crop season. Less significant effect of long-term (12 years) gypsum application on Ca^{2+}

Table 5 Levels of mineral nitrogen ($NH_4^+ + NO_3^-$) for 2017 and 2018 crop seasons of a no-till Typic Hapludox in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates top-dressed at tillering (N_0, N_{50}, and N_{100} refers to 0, 50, and 100 kg ha^{-1} of N) in a no-till Typic Hapludox in Guarapuava, Paraná state, Brazil

Depth (m)	Gypsum (Mg ha^{-1})	Mineral-N (mg dm^{-3})					
		2017			2018		
		N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}
0–0.1	0	9 b	27 a	25 a	10 b	13 b	16 a
	3	11 c	22 a	16 b	6 b	11 b	16 a
	6	13 c	19 a	16 b	7 b	9 b	11 a
	9	13 b	14 a	15 a	8 b	11 b	14 a
	12	11 b	15 a	15 a	7 b	9 a	9 a
Linear (R^2)		0.25^{*}	0.84^{***}	0.56^{***}	0.05^{ns}	0.53^{***}	0.37^{**}
Quadratic (R^2)		0.63^{***}	0.91^{**}	0.81^{**}	0.45^{***}	0.66^{***}	0.75^{**}
0.1–0.2	0	12 b	15 a	14 a	8 a	8 a	8 a
	3	10 c	13 b	17 a	8 a	8 a	8 a
	6	13 a	13 a	11 b	7 a	8 a	8 a
	9	13 a	13 a	13 a	5 a	4 a	5 a
	12	10 b	11 b	15 a	9 b	6 c	10 a
Linear (R^2)		0.00^{ns}	0.43^{**}	0.05^{ns}	0.04^{ns}	0.50^{***}	0.03^{ns}
Quadratic (R^2)		0.21^{*}	0.42^{**}	0.14^{ns}	0.29^{*}	0.51^{***}	0.25^{ns}
0.2–0.4	0	15 b	17 a	13 b	9 a	7 a	7 a
	3	8 c	12 b	16 a	9 a	10 a	9 a
	6	13 a	15 a	13 a	6 a	5 a	6 a
	9	10 a	11 a	11 a	7 a	5 b	5 b
	12	8 c	13 b	17 a	9 a	8 a	8 a
Linear (R^2)		0.36^{**}	0.28^{*}	0.02^{ns}	0.02^{ns}	0.02^{ns}	0.04^{ns}
Quadratic (R^2)		0.35^{*}	0.48^{***}	0.15^{ns}	0.51^{**}	0.07^{ns}	0.07^{ns}
0.4–0.6	0	12 b	14 a	11 b	6 b	6 b	10 a
	3	9 b	11 a	10 ab	5 a	6 a	7 a
	6	11 a	12 a	12 a	7 b	5 b	9 a
	9	10 b	11 ab	12 a	6 b	4 b	8 a
	12	9 b	11 a	11 a	7 b	5 b	10 a
Linear (R^2)		0.33^{**}	0.29^{*}	0.06^{ns}	0.63^{***}	0.03^{ns}	0.70^{***}
Quadratic (R^2)		0.34^{**}	0.36^{***}	0.07^{ns}	0.64^{***}	0.05^{ns}	0.94^{**}
0.6–0.8	0	11 a	8 b	11 a	5 b	8 a	6 b
	3	9 c	11 b	16 a	5 b	4 b	7 a
	6	14 a	11 b	12 b	6 a	6 a	5 a
	9	9 c	11 b	14 a	6 a	5 a	3 b
	12	14 a	14 a	9 b	8 a	2 c	5 b
Linear (R^2)		0.09^{ns}	0.75^{***}	0.15^{ns}	0.58^{***}	0.60^{***}	0.30^{*}
Quadratic (R^2)		0.12^{ns}	0.76^{**}	0.57^{**}	0.79^{**}	0.61^{*}	0.42^{**}

Lowercase letters compare the N-fertilization averages within each crop season. ***, **, * and ^{ns} denote significant values at $p \leq 0.001$, $p \leq 0.01$, $p \leq 0.05$, and non-significant values, respectively. Additional information on the mineral-N level is presented in Fig. S3

levels in the upper soil layers is corroborated by Fontoura et al. (2019). In fact, layers up to 0.4 m from surface presented higher Ca^{2+} variability and data dispersion (lower R^2), which is consistent to the cumulative effect of different species with varying rooting depths, as well as different capacities of nutrient extraction and recycling in the cropping system in the area.

Evaluating the long-term effect of gypsum allows us to estimate with greater precision the benefits and viability of its use in soil fertility management. According to our results, even if the gypsum was applied at a rate of 3 Mg ha^{-1}, the trend of a higher level of Ca^{2+} in the subsurface remains past 6 to 7 years the last gypsum application. In addition, Ca^{2+} levels remained above the general critical level of 2 cmol$_c$ dm^{-3} (SBCS/NEPAR 2019) in all treatments, with exception of control treatment in subsurface. N-fertilization had no effect on soil Ca^{2+} levels, which also corroborates the results of Vicensi et al. (2020a).

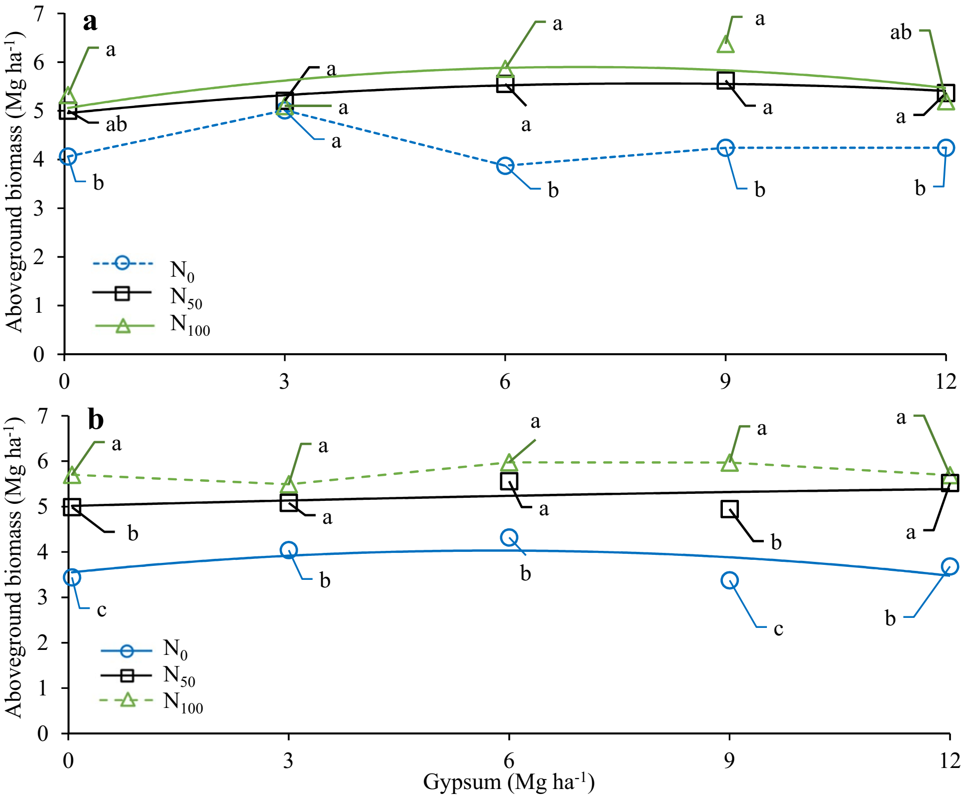

Fig. 2 Black oat forage yield in 2017 (**a**) and 2018 (**b**) crop seasons, in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates (N_0, N_{50}, and N_{100} corresponding to 0, 50, and 100 kg ha^{-1} of N) top-dressed at tillering, in a no-till field in Guarapuava, Paraná state, Brazil. Lower letters in the fertilizer N rates classify the averages by the Tukey test at 5% probability. In gypsum rates, the solid line represents the regression trendline, whereas the dashed line was used with non-significant regression. Results of analysis of variance of forage yield are presented in Table S1

Table 6 Contents of nitrogen (N), potassium (K), calcium (Ca), and magnesium (Mg) in black oats at bolting stage in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates top-dressed at tillering, under no-tillage Typic Hapludox during the 2017 and 2018 crop seasons in Guarapuava, Paraná state, Brazil

Gypsum (Mg ha^{-1})	N (kg ha^{-1})		K (kg ha^{-1})		Ca (kg ha^{-1})		Mg (kg ha^{-1})	
	2017	2018	2017	2018	2017	2018	2017	2018
0	118	115	108	105	21	21	10	9.7
3	139	135	133	129	25	24	12	11
6	141	142	131	132	28	28	11	11
9	133	117	147	119	30	26	11	9.8
12	131	133	121	116	27	27	10	9.6
Linear (R^2)	0.01^{ns}	0.00^{ns}	0.00^{ns}	0.00^{ns}	0.12^{**}	0.10^{**}	-	-
Quadratic (R^2)	0.08^{ns}	0.04^{ns}	0.00^{ns}	0.00^{ns}	0.06^{*}	0.06^{*}	-	-
N (kg ha^{-1})								
0	100 b	86 c	116 a	91 c	24 b	19 b	9.6 b	7.8 b
50	149 a	135 b	131 a	119 b	28 a	27 a	11 a	11 a
100	148 a	156 a	137 a	150 a	28 a	29 a	12 a	12 a
Gypsum (G)	$\mathbf{0.01}^{*}$	$\mathbf{0.01}^{*}$	$\mathbf{0.02}^{*}$	$\mathbf{0.02}^{*}$	$\mathbf{0.03}^{*}$	$\mathbf{0.00}^{***}$	0.25^{ns}	0.12^{ns}
N	$\mathbf{0.00}^{***}$	$\mathbf{0.00}^{***}$	$\mathbf{0.05}^{*}$	$\mathbf{0.00}^{***}$	$\mathbf{0.02}^{*}$	$\mathbf{0.00}^{***}$	$\mathbf{0.01}^{**}$	$\mathbf{0.00}^{***}$
G×N	0.62^{ns}	0.70^{ns}	0.53^{ns}	0.52^{ns}	0.90^{ns}	0.56^{ns}	0.70^{ns}	0.62^{ns}
CV_G(%)	10	15	19	15	22	14	22	16
CV_N(%)	11	14	22	12	21	16	20	18

Bold numbers are statistically significant. Lowercase letters compare the N-fertilization averages within each crop season by the Tukey test at 5%. ***, **, * and ^{ns} denote significant values at $p \leq 0.001$, $p \leq 0.01$, $p \leq 0.05$, and non-significant values, respectively

Gypsum is commonly reported to increase soil levels of Ca^{2+} and $S\text{-}SO_4^{2-}$ and leach or mobilize Mg^{2+} from the surface to the subsurface (Vicensi et al. 2016; 2020a), and in our study, the Mg^{2+} level in the soil presented a decrease pattern up to 0.8 m depth due to the increase in gypsum rate. The initial Mg level prior to gypsum installment (2009)

Table 7 Contents of phosphorus (P) and sulfur (S) in black oats at bolting stage in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates (N0, N50, and N100 refers to 0, 50, and 100 kg ha−1 of N) top-dressed at tillering, under no-tillage Typic Hapludox during the 2017 and 2018 crop seasons in Guarapuava, Paraná state, Brazil

Gypsum (Mg ha^{-1})	P (kg ha^{-1})					
	2017			2018		
	N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}
0	16 ab	13 b	17 a	12 b	14 b	18 a
3	13 a	15 a	14 a	14 a	16 a	15 a
6	19 ab	22 a	17 b	15 b	20 a	18 a
9	20 ab	23 a	19 b	14 b	15 b	22 a
12	15 b	26 a	23 a	13 c	27 a	23 b
Linear (R^2)	0.00^{ns}	0.29^{*}	0.35^{**}	0.04^{ns}	0.45^{**}	0.41^{**}
Quadratic (R^2)	0.13^{ns}	0.29^{*}	0.37^{*}	0.27*	0.48^{*}	0.49^{*}
Gypsum (G)	**<0.001*****			**<0.001*****		
N	**<0.001*****			**<0.001*****		
G × N	**<0.001*****			**<0.001*****		
CV_G(%)	16			11		
CV_N(%)	12			9		

Gypsum (Mg ha^{-1})	S (kg ha^{-1})					
	2017			2018		
	N_0	N_{50}	N_{100}	N_0	N_{50}	N_{100}
0	48 a	55 a	57 a	33 b	45 b	63 a
3	59 a	42 a	47 a	41 a	39 a	35 a
6	68 a	64 a	58 a	63 a	64 a	60 a
9	57 b	61 b	98 a	48 b	58 ab	67 a
12	60 c	103 b	142 a	44 c	123 b	141 a
Linear (R^2)	0.04^{ns}	0.35^{**}	0.59^{***}	0.11^{ns}	0.62^{***}	0.48^{***}
Quadratic (R^2)	0.12^{ns}	0.49^{*}	0.74^{**}	0.49^{*}	0.80^{**}	0.81^{**}
Gypsum (G)	**0.002****			**<0.001*****		
N	**0.003****			**<0.001*****		
G × N	**<0.001*****			**<0.001*****		
CV_G(%)	35			26		
CV_N(%)	28			15		

Bold numbers are statistically significant. Lowercase letters compare the N-fertilization averages within each crop season by the Tukey test at 5%; *** and ** denote significant values at $p \leq 0.001$ and $p \leq 0.01$, respectively

was 2.5 cmol$_c$ dm^{-3} up to 0.2 m depth (Table 1), but after 6 to 7 years of the last gypsum application, the Mg level decreased below the general critical level of 1 cmol$_c$ dm^{-3} (SBCS/NEPAR 2019) in all treatments and up to 0.8 m. Such information evidences that in long-term gypsum application, Mg^{2+} levels should be monitored and, if necessary, replaced. In our study, it must be considered that until this moment, besides being leached after the reaction with gypsum S-SO_4^{2-}, Mg^{2+} was also exported through the harvested products (grains, forage) and not replenished.

Among the measures to compensate for the reduction of Mg^{2+} on the surface soil layers (0.0–0.2 m), and to keep the Mg^{2+} level above its critical level, magnesium thermophosphate can be used in sowing fertilization, and when liming is necessary, preference should be given to the use of dolomitic lime (SBCS/NEPAR 2019). The lower levels of Mg^{2+} due to the gypsum application are related to a greater potential of Mg^{2+} leaching, and the lowest level of these nutrient in G_6 compared to higher gypsum rates are possibly due this rate achieved the maximum yields of forage and grains in previous crops along the years, especially in Poaceae species (Vicensi et al. 2016; 2020a), which means that this rate caused a greater depletion of soil nutrients over time.

The levels of S-SO_4^{2-} increased in response to gypsum rates in all evaluated soil layers, but particularly in depth. In all treatments, the S-SO_4^{2-} level remained above the general critical level of 3 and 9 mg dm^{-3} in the 0.0–0.2 m and 0.2–0.4 m layers (SBCS/NEPAR 2019), respectively. The soil where this study was conducted has been under no-tillage since 2005, which in part explain the high soil organic matter content of this field site as reported by Michalovicz et al. (2019), consistent to the high levels of S-SO_4^{2-} and mineral-N in the soil. Also, the soil was sampled 7 to 10 days after N topdressing during black oat cultivation, so the high levels of mineral-N in the soil profile

Table 8 Apparent nitrogen recovery efficiency in black oats at bolting stage in response to the residual effect of gypsum rates (applied between November 2009 and November 2011) and nitrogen rates (N50 and N100 refers to 50 and 100 kg ha−1 of N) top-dressed at tillering, under no-tillage Typic Hapludox during the 2017 and 2018 crop seasons in Guarapuava, Paraná state, Brazil

Apparent nitrogen recovery efficiency ($kg_{[N\ content]}\ kg_{[N\ applied]}^{-1}$)								
Crop seasons	2017				2018			
Gypsum ($Mg\ ha^{-1}$)	N_{50}		N_{100}		N_{50}		N_{100}	
0	1.08	a	0.55	b	1.08	a	0.82	b
3	0.86	a	0.35	b	0.98	a	0.65	b
6	0.99	a	0.51	b	1.25	a	0.62	b
9	0.81	a	0.55	b	0.87	a	0.8	a
12	1.14	a	0.43	b	0.75	a	0.65	a
Linear (R^2)	0.00^{ns}		0.00^{ns}		0.19^{ns}		0.03^{ns}	
Quadratic (R^2)	0.22^{ns}		0.05^{ns}		0.30^{*}		0.06^{ns}	
ANOVA (p value)								
Gypsum (G)	0.324^{ns}				0.267^{ns}			
Nitrogen (N)	**<0.0001*****				**<0.0001*****			
G × N	**0.043***				**0.013***			
CV_G (%)	29				28			
CV_N (%)	17				18			

Bold numbers are statistically significant. Lowercase letters in the line within each crop season compare the averages by the Tukey test at 5%; ***, * and ^{ns} denote significant values at $p \leq 0.001$ and $p \leq 0.05$ and non-significant values, respectively

confirms the adequacy of the N-fertilization recommendation used (SBCS/NEPAR 2019). In warmer environments and in soils with lower levels of organic matter in southern Brazil, there may be a response to higher N-fertilization rates than those used in this study (Ribeiro et al. 2020).

The mineral-N levels presented lower values in 2018 compared to 2017 crop season. The water balance showed greater water availability in 2018 at the time of N-application which possibly accounted for higher N leaching (N losses) compared to 2017. Other reason for this difference is the cumulative effect of crop sequence, considering that only Poaceae species were cultivated during the experimental period (maize/oat in 2017, maize/oat in 2018). The crop residues of Poaceae species have a higher carbon:nitrogen (C:N) ratio, which increases N immobilization by microorganisms (organic-N) during decomposition (Sprunger et al. 2019), so less N is mineralized (mineral-N) and released to the soil.

In 2017, gypsum decreased mineral-N in layers up to 0.6 m. As gypsum enhanced the $S\text{-}SO_4^{2-}$ levels in the soil, $S\text{-}SO_4^{2-}$ competes with $N\text{-}NO_3^{-}$ for the positive charge sites in soil colloids, which may have resulted in less adsorbed $N\text{-}NO_3^{-}$ and, therefore, lower mineral-N levels in the soil, which may have contributed to higher N content in the forage due to gypsum. In 2018, under higher water availability condition, the decrease effect on mineral-N over soil layers was less evident over gypsum rates, reflecting different conditions of root and above ground growth and, therefore, contrasting capacities of N extraction/depletion in the soil.

The N-fertilization increased soil mineral-N because urea is rapidly hydrolyzed in the soil by the urease enzyme, forming ammonia ($N\text{-}NH_3$) and then ammonium ($N\text{-}NH_4^{+}$), which through bio-oxidation will be sequentially transformed into nitrite ($N\text{-}NO_2^{-}$) and nitrate ($N\text{-}NO_3^{-}$), consequently increasing soil mineral-N levels (Sigurdarson et al. 2018). In 2018, there was a lower N availability in the soil compared to 2017 crop season and even lower under N fertilization rates. This fact probably reflects the greater N depletion in the soil due crop response to N-fertilization as observed in Black oat forage yield and lower N content in the forage without N fertilization.

The forage yield was, on average, 13% lower in 2018 crop season compared to the 2017, which is consistent to the lower soil mineral-N in 2018, as well as the lower accumulated degree days (− 10.4%) and the lower accumulated solar radiation (− 7.6%) in 2018 compared to 2017.

Fertilization with N enhances black oat vegetative growth, increases carbon and nitrogen inputs in the biomass (Ribeiro et al. 2020), and, consequently, increases black oat yield of forage and residual biomass (Vicensi et al. 2021a,b).). Preceded by maize, forage yield over seasons increased, on average, by 32% and 41% with N_{50} and N_{100}, respectively. Previously in the same experimental area but preceded by soybean, forage yield of the same black oat cultivar was reported to increase, on average, by 9% and 15% with N_{50} and N_{100}, respectively (Vicensi et al. 2020b). The greater response to N fertilization in black oat cultivated after maize, when compared to black oat cultivated after soybean, may be due the N mineralized from the low C:N ratio residue of soybean, as well as the N in the soil from "sparing effect" of a N-fixing crop like soybean increases soil N availability and reduces fertilizer-N dependence (Guinet et al. 2020), lowering black oat response to N-fertilization as discussed by Vicensi et al. (2020b). After maize, high C:N ratio residues added to the soil, with values ranging from 30:1 for roots (Ordóñez et al. 2020) to 60:1 for aboveground part (Sprunger et al. 2019), increases N immobilization by soil microorganisms, lowering the availability of soil mineral-N for plants, which will be more dependent on N-fertilization as evidenced in our results.

The official N-fertilization recommendation for forage black oat (winter forage grasses) in subtropical Brazil does not consider the preceding crop. In the southernmost Brazilian states of Rio Grande do Sul and Santa Catarina, only the soil organic matter levels are considered (SBCS/NRS 2016). Bordering Santa Catarina, Paraná State, has a recommendation based on the expected forage dry matter yield (SBCS/NEPAR 2019), and ranges are 35–125 $kg_{[N]}$ ha^{-1} for grazing and 75–250 $kg_{[N]}$ ha^{-1} for silage and hay production. In this study, as previously in this experiment, black oat was top-dressed up to 100 $kg_{[N]}$ ha^{-1} and cut once at pre-flowering is suitable for forage yield and quality.

As black oat forage yield has been reported to increase in response to N-fertilization (Joris et al. 2016), with rates above 100 $kg_{[N]}$ ha^{-1} (Ribeiro et al. 2020), which was the highest rate tested in this study, and considering that the experiment site is in a region where winters are cold, maize is an important summer crop in rotation with soybean in the prevailing grain production system; it is possible that higher forage yields would be achieved if N supply was higher, so further studies should be carried out to help refine N-fertilization recommendation for forage black oat in similar subtropical conditions.

Previously in this same experiment, gypsum significantly increased forage yield of black oat 4 to 5 years past its application (Vicensi et al. 2020b; 2021a), as well as grain yield of other Poaceae a long time before (Vicensi et al. 2016; 2020a; 2020b). Unlikely, in this study past 6 to 7 years from the last application, the residual effect of gypsum did not affect forage yield independently of interaction with N and evidence that gypsum lost the capacity to explain forage yield changes in this long term. Past 5 years after the last gypsum application, Costa and Crusciol (2016) found no effect in black oat biomass in response to a cumulative rate of 4 Mg ha^{-1} of gypsum. These are indications about the period of significant residual effect of gypsum alone, which is an important indication for field recommendation.

Soil Mg^{2+} levels in 2017 and 2018 were mostly below the general critical level of Mg^{2+} (1.0 $cmol_c$ dm^{-3}). The drop from the initial level of 2.5 $cmol_c$ dm^{-3} is justified by the fact that no Mg^{2+} replenishment to the soil was intentionally performed since 2009, but also by the fact that gypsum leached Mg^{2+}. So the lower gypsum effect on forage yield in this last period of the study may evidence that the soil critical level of Mg^{2+} should be close to 1.0 $cmol_c$ dm^{-3} for black oat, and without replenishing soil Mg^{2+}, the benefits of gypsum to crop yields are hindered. In another study, under low levels of Mg^{2+} in the soil, the application of 4 Mg ha^{-1} of gypsum had no effect on black oat biomass, but when associated with the application of dolomitic lime, which is a Mg^{2+} source, the crop biomass increased by 2.9-fold (Costa and Crusciol 2016).

In both crop seasons, the contents of N and K in the forage were affected by long-term gypsum and had an increase pattern in response to the rates applied 6–7 years before. If the higher Ca content in the forage is related to the Ca^{2+} increase in the soil due to gypsum, meanwhile gypsum reduced mineral-N levels in the soil up to 0.6 m depth, possibly the gypsum effect on greater black oat root growth (Vicensi et al. 2020b) favored water and nutrients absorption, as well as a depletion effect on soil mineral-N over time.

Contents of P and S also increased in response to gypsum, meanwhile S-SO_4^{-2} availability in the soil in depth increased due to gypsum, which evidence the indirect benefit of gypsum to nutrient absorption through better root growth, which was already reported for black oat (Vicensi et al. 2020b), and for maize (Caires et al. 2016). The absorption of Mg, as an exception, was not benefited by the effect of gypsum. In fact, gypsum decreased Mg^{2+} levels in the soil, and it may be the main limiting factor for further gypsum benefits to crops along time after application, as evidenced by the lack of gypsum effect in forage yield in the present period of study compared to the benefits observed previously in this same experimental area (Vicensi et al. 2020b, 2021a).

N-fertilization increased the contents of N, K, Ca, Mg, P, and S in both crop seasons, in a proportion similar to or greater than the consequent increase in forage yield. Increasing forage yield and nutrient content are directly beneficial to the herd to be fed, but also to the soil and to the successor crops after oat cycle is finished. N-fertilization may also increase soil cover through black oat regrowth and increase the potential for nutrient cycling to the next crop, as discussed by Vicensi et al. (2021a), what is particularly beneficial if succeeding crop has a shallow root system (Fan et al. 2016).

The roots of black oat reach 0.8 m depth (Vicensi et al. 2020b), so the crop is able to uptake nutrients from subsurface soil layers and to recycle them to the next crop through the residue decomposition. The highest rate of mineralization and nutrient release from black oat residues occurs close to 30 days after deposition (Ferreira et al. 2014), and the calculated half-life times for nutrients released from black oat residues are 15, 10, 9, 8, and 9 days for N, P, K, Ca, and Mg, respectively (Ferreira et al. 2014).

As a rule, apparent N recovery efficiency (RE) did not differ between the gypsum rates within both crop seasons, what is consistent to the general absence of gypsum effect in black oat forage yield and to the fact that N content in the forage fitted no linear or quadratic model. Gypsum associated with ammonia nitrate (NH_4NO_3) has been reported to improve N use efficiency in maize cultivated under no-till, as a result of gypsum effect mitigating subsurface acidity (Caires et al. 2016). In our study, gypsum almost had no effect on nitrogen RE possibly because Ca^{2+} was above the critical level (2 $cmol_c\ dm^{-3}$) and Al^{3+} levels were low in our soil, and gypsum was applied much time before the evaluation period. In addition, black oat was harvested in pre-flowering in our study, when N demand by the plants is lower compared to maize at maturation as evaluated by Caires et al. (2016), other possible limit to gypsum effects on nitrogen RE by black oat. Caires et al. (2016) reported that gypsum can improve maize root growth, contributing to increase RE and to reduce NO_3^- leaching (Caires et al. 2016; Bossolani et al. 2020). Increased root growth in response to gypsum was also reported to other Poaceae crops (Costa et al. 2018), including black oat (Vicensi et al. 2020b).

Doubling N fertilization from N_{50} to N_{100} did not increase crop N uptake proportionally (Table 6), but strongly decreased RE (Table 8). Higher N fertilization rate does not always increase nutrient content, but reduces N fertilizer RE and increases post-harvest soil N levels, which increases the risk of N losses, either by leaching, volatilization, and denitrification (Greer and Pittelkow 2018). The same effect was observed in wheat, where the higher the rate, the lower the efficiency of N used (Todeschini et al. 2016; Caires et al. 2022), as well as in other winter grasses (Vogeler et al. 2020).

In our study, 30 days prior to black oat cutting in the 2017, there was more intense water restriction compared to 2018, which is possibly associated with a lower average RE observed in 2017 (0.48 kg kg^{-1}) compared to 2018 (0.71 kg kg^{-1}). The main form of contact between the root and the mineral-N is through mass flow (Taiz et al. 2017), which indicates that water availability is necessary for the nutrient to be properly absorbed by the plants.

5 Conclusions

Our hypothesis was not confirmed, and no synergistic effect was observed in forage yield and apparent N recovery by black oat in response to the long-term effect of gypsum associated with nitrogen fertilization.

Although Ca^{2+} and SO_4^{2-} levels in subsurface soil were still higher past 6–7 years since last gypsum application, Mg^{2+} decreases in the soil profile, especially with the application of higher rates (9 and 12 Mg ha^{-1}), what may have limited the performance of black oat. Soil mineral-N was also decreased by gypsum, whereas N-fertilization replenished those levels.

N-fertilization improved forage quality through higher contents of N, P, K, Ca, Mg, and S. Gypsum also improved the forage contents of N, K, Ca, and the contents of P and S when associated with N-fertilization.

The rate of 50 $kg_{[N]}\ ha^{-1}$ in black oat enabled a more assertive use of fertilizer N, as evidenced by the greater apparent N recover compared to 100 $kg_{[N]}\ ha^{-1}$ in a field previously cultivated with maize. An additive effect was achieved with at least 3 Mg ha^{-1} rate of long-term applied gypsum in terms of forage yield and macronutrient composition in a black oat grown in a subtropical environment without severe drought.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s42729-022-00899-5.

Acknowledgements We acknowledge the *Universidade Estadual do Centro-Oeste (Paraná)* for supporting this research, the staff of the *Departamento de Agronomia* and the *Laboratório de Solos e Nutrição de Plantas*, especially to Luis Henrique de Lima and Ana Paula Vantroba.

Funding This study is funded by *Coordenação de Aperfeiçoamento de Pessoal de Nível Superior* (Coordination for the Improvement of Higher Education Personnel, CAPES—Brazil) – Financial code 001; *Conselho Nacional de Desenvolvimento Científico e Tecnológico* (National Counsel of Technological and Scientific Development, CNPq—Brazil)—Grant no. 312024/2018–5; and *Fundação Araucária* (Araucária Foundation from Paraná State—Brazil).

Declarations

Conflict of Interest The authors declare no competing interests.

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