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Effects of Gypsum Application Rate and Frequency on Corn Response to Nitrogen

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ABSTRACT

The effects of gypsum as a soil amendment and its interaction with nitrogen (N) applications in corn (*Zea Mays* L.) production are largely unknown. A 4-yr field study was conducted in Ohio, USA to evaluate (i) the effects of gypsum rates and rate frequencies at varying fertilizer N rates on corn grain yields, ear-leaf and grain nutrient concentrations, and on soil nutrients, and (ii) the interactive effects between N and gypsum applications. A split-plot experimental design was used with N rates (0, 84, 168, and 252 kg N ha^{-1}) as the main plot factor and either gypsum application rate (0, 1.1 and 2.2 Mg ha^{-1}) or application rate frequencies (i.e., 1.1 Mg ha^{-1} annually, 2.2 Mg ha^{-1} bi-annually, and 4.4 Mg ha^{-1} once every four years) as subplot factors. Overall, corn yield response to gypsum applications was inconsistent. A positive corn yield response to gypsum application occurred only in 2017 at the Northwest site. Gypsum applied at 4.4 Mg ha^{-1} significantly decreased yields at the Northwest site in 2017 and at the Wooster site in 2016 suggesting that the best management strategy may be to apply lower gypsum rates either annually or biannually. Gypsum applications did not improve corn response to N at either study site but significantly increased tissue, grain and extractable soil S concentrations with concentrations decreasing as the application frequency decreased. This suggests that where S deficiencies may occur, an annual rate of 1.1 Mg ha^{-1} or a bi-annual application of 2.2 Mg ha^{-1} would increase S availability.

Core Ideas

- Corn response to N at different gypsum application rates and frequencies was evaluated.
- Gypsum did not improve corn response to N at any of the study sites.
- Corn yield response to gypsum applications was very inconsistent and site-specific.
- Frequent gypsum applications at lower rates were better than a one-time large application.

Gypsum is commonly used as a soil amendment and as a fertilizer in agriculture. In fact, it is one of the earliest forms of fertilizer used in the United States, and its use dates back more than 200 yr (Wallace, 1994). Gypsum is an excellent source of calcium (Ca) and sulfur (S) for plant nutrition, as Ca^{2+} and SO_4^{2-} ions become readily available in soil solution after its application (Shainberg et al., 1989; Chen and Dick, 2011). Recently, there has been an enhanced interest in using gypsum as a S source due to a potential for S deficiency in crops resulting from reduced S inputs (Chen et al. 2005, 2008; Buckley and Wolkowski, 2012; Kim et al., 2013; Steinke et al., 2015; Sutradhar et al., 2017).

Gypsum as a soil amendment or conditioner, is known to provide a multitude of benefits by promoting soil aggregation and enhancing soil physical structure. Gypsum can reduce soil dispersion, prevent soil crust formation, promote better seedling germination, facilitate better water infiltration and soil aeration, improve crop water relations, increase soil drainage, reduce soil erosion and runoff, and also improve water quality by decreasing phosphorus (P) losses from soils (Shainberg et al., 1989; Wallace, 1994; Amézketa, 1999; Amezketa et al., 2005; King et al., 2016; Kost et al., 2018). Gypsum application aids in the alleviation of aluminum (Al^{3+}) toxicity and promotes better plant growth in soils affected by subsoil acidity (Toma et al., 1999). Gypsum can, therefore, favorably modify soil physical and chemical properties to provide better growing environments for plants. This could, in turn, facilitate plant root exploration into greater soil volumes and to deeper depths of the soil profile resulting in better water and nutrient use efficiency and might reduce nutrient input needs (e.g., N) (Dick et al., 2006; Watts and Dick, 2014).

Given its wide range of benefits, producers are interested to incorporate gypsum as one of the management practices to improve agricultural production and sustainability. The benefits associated with gypsum applications are, however, both short- and long-term and depend on the purpose for which it is being used. Nutritional benefits (i.e., S fertilization) associated with gypsum are short-term (1–2 yr), direct and require lower application rates. Changes in soil physical and chemical properties require more time (>3 yr) and often repeated gypsum applications at higher rates, to produce any yield benefits (Watts and Dick, 2014). Annual application rates can therefore range from 100 kg ha^{-1} when gypsum is used as a nutrient source to several megagrams per hectare if used as a soil conditioner (Chen and Dick, 2011).

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Recommended gypsum application rates for improving soil properties in Ohio and in other parts of the United States range from 1.1 to 4.5 Mg ha^{-1} (NRCS, 2015). Effects of gypsum application as a nutrient source at lower rates on corn grain yields are well studied, but gypsum applied at higher rates and the interactive effects among gypsum rate and nitrogen rate on corn yields are not as well understood. Also, it is unclear whether applying gypsum annually at lower rates versus applying the same total amount via a one-time higher application rate, affects the results observed when applying gypsum as a soil amendment. It is important to optimize gypsum applications as a large proportion of the cost of gypsum is tied up in transportation. Less frequent applications of gypsum but at higher rates would be economically more attractive. As much as gypsum is beneficial, economics will be the ultimate driving factor for gypsum to be included as a management practice as its transportation and spreading costs keep rising (Kost et al., 2014).

Nitrogen is a key primary plant nutrient that plays a vital role in crop yields and enhanced crop quality (Marschner, 1995). However, when N inputs exceed crop demands, farmer profitability is compromised and water quality degradation often results from offsite movement of nitrate (Jaynes et al., 2001; Scharf et al., 2005; Helmers et al., 2012; McIsaac, 2016). Previous studies have found that gypsum application (i.e., S fertilization) can improve N use and result in a positive corn yield response at lower N fertilization rates due to regulatory effects of N and S on each other (Weil and Mughogho, 2000; Chen et al., 2005; Steinke et al., 2015). These authors, therefore, concluded that gypsum applications may even result in lowering the recommended rates of N fertilization in corn. However, in these studies, gypsum was applied as a S source at lower application rates rather than at higher soil amendment rates. Whether gypsum applied as a soil amendment at higher agronomic rates but less frequently, rather than low rates applied more frequently when gypsum is used primarily as a S source, results in improved N use in corn is an important research question that has not been evaluated previously.

Therefore, a 4-yr field study was conducted with an overall goal to test the effects of a one-time higher gypsum application rate versus more frequent lower application rates on corn grain yield, crop nutrient uptake and soil chemical properties and if gypsum applied at higher rates leads to any improved N uptake in continuous corn. Specific objectives were to evaluate (i) the direct effects of gypsum rates and rate frequencies at varying fertilizer N rates on corn grain yields, ear leaf and grain nutrient concentrations, and on soil extractable nutrients; and (ii) the interactive effects between fertilizer N and gypsum applications.

MATERIALS AND METHODS

Site and Experimental Setup

Two field trials were established in 2014 in Ohio, USA and continued until 2017. One site is located near the city of Bowling Green, OH (Wood County) on a Hoytville clay loam (fine, illitic, mesic Mollic Epiaqualf) at the Northwest Agricultural Research Station (41°13′20″ N, 83°45′37.7″ W). The other site is located near the city of Wooster, OH (Wayne County) on a Canfield silt loam (fine-loamy, mixed, active, mesic Aquic Fragiudalf) at the Ohio Agricultural Research and Development Center (40°45′27.2″ N, 81°53′52″ W). From here on, they are designated as Northwest and Wooster locations. Soils (0–20 cm) at Northwest location have a cation exchange capacity (CEC) of 17.1 $cmol_c$ kg^{-1}, a pH of 6.33 and an organic matter content of 21.3 g kg^{-1}. Soil extractable Ca, Mg, K and S concentrations were 2317, 349, 168, and 15.2 mg kg^{-1}, respectively, at the Northwest site. Similarly, soils (0–20 cm) at the Wooster site have a CEC of 7.18 $cmol_c$ kg^{-1}, a pH of 6.58 and an organic matter content of 12.6 g kg^{-1}. Soil extractable Ca, Mg, K and S concentrations at this site were 1140, 204, 56, and 12.1 mg kg^{-1}, respectively. Proportions of sand, silt and clay were 22, 30, 48% at Northwest and 16, 59, 25% at Wooster, respectively (Walia and Dick, 2018).

Two different long-term gypsum application strategies were tested in this study over a 4-yr period: a) gypsum applied at different rates on an annual basis and b) the same amount of gypsum applied but at different rates and frequencies over a 4-yr cycle. A split plot, randomized block experimental design was used at each site with three replications. The main plot factor consisted of N rates and the subplot factor consisted of either the gypsum rate or gypsum rate frequency. The two different gypsum applications were spatially integrated as they both received the same N rate treatments. So, each block was divided into four main plots, which randomly received four nitrogen rates (0, 84, 168, and 252 kg N ha^{-1}) and each main plot was equally split into six subplots that randomly received the three gypsum rates and three gypsum rate frequency treatments. Each main N plot measured 6 m by 27 m and each individual gypsum subplot measured 3 m by 9 m. The three gypsum rate treatments included 0 Mg ha^{-1} (control), and 1.1 Mg ha^{-1}, and 2.2 Mg ha^{-1} of gypsum, with 1.1 and 2.2 Mg ha^{-1} applied every year continuously for 4 yr. The three gypsum rate frequency treatments included 1.1 Mg ha^{-1} every year (annual), 2.2 Mg ha^{-1} every 2 yr (bi-annual) and 4.4 Mg ha^{-1} every 4 yr (4-yr), respectively. Thus, over a 4-yr cycle, the same total amount of gypsum was applied to each frequency treatment. Application rates were based on typical grower practices in the region and guided by the Natural Resource Conservation Service, Ohio Conservation Practice Standard (NRCS, 2015). Due to space limitations, the zero N rate main plot treatment received only three gypsum treatments (no gypsum, gypsum applied at 1.1 Mg ha^{-1} annually and gypsum applied at 2.2 Mg ha^{-1} every 2 yr), which made this an unbalanced split plot design.

Continuous corn was grown under no-till conditions for this study starting in 2014 at both sites. Each subplot had four rows of corn planted with a row spacing of 76 cm. Corn was planted in early May in all years except in 2014 at the Northwest and Wooster sites and in 2017 at the Wooster location, due to unusual wet weather or establishment issues, which delayed planting. The gypsum ($CaSO_4 \cdot 2H_2O$) used was a by-product of the flue gas desulfurization (FGD) process and was surface applied by broadcast to individual subplots following the rate and frequency treatments mentioned above. Whenever there was a gypsum application, it was done as a one-time pre-plant application during the spring season just before planting of corn. Nitrogen fertilizer was applied as a single side-dress application at the V4-V6 growth stage in the form of urea-ammonium nitrate using a Coulter injector at rates equivalent to 0, 84, 168, and 252 kg N ha^{-1}. Fertilization with P and K was based on recommendations given in Vitosh et al. (1995) and management of weeds, insects and diseases followed established cultural protocols given in The Ohio State University recommendations for the agronomic production of corn (Thomison et al., 2005; Loux et al., 2017). After physiological maturity, corn grain was harvested from the middle two rows of the four-row plots using a small plot combine. Corn yields (2014–2017) were calculated using the grain weight and moisture content from the combine and were adjusted to 155 g kg^{-1} moisture content.

Sample Collection

Tissue and grain samples were collected only in 2017 at the end of one complete 4-yr cycle. Corn ear-leaf tissue samples were collected at the beginning of the reproductive phase (R1) for nutrient analysis. Corn ear-leaves (8 $plot^{-1}$) and representative grain samples during harvesting were collected from the two middle rows of each experimental plot and bagged. Ear-leaf tissue and grain samples were dried in a forced-air oven for 72 h at 65°C, ground using a Wiley stainless steel mill (Thomas Scientific, Swedesboro, NJ) to pass through a 1-mm mesh screen and microwave digested using a nitric acid-per chloric acid mixture and analyzed for Ca, Mg, K, and S using inductively coupled plasma atomic emission spectroscopy (ICP–AES) (Isaac and Johnson, 1985). Ear-leaf tissue and grain N concentrations were determined by the Dumas combustion method (AOAC International, 2002).

Soil samples were also collected at the end of the growing season in 2017 from the surface 0- to 20-cm of all subplots. Eight soil cores (2.5 cm diam.) were randomly sampled between the plant rows from each subplot and were composited as a single sample. Composite soil samples were passed through an 8-mm sieve, mixed and air-dried. Soils were then ground to pass through a 2-mm sieve to determine pH, CEC, organic matter and Mehlich III extractable Ca, Mg, K and S using recommended chemical soil test procedures for the North Central Region (NCERA-13). Soil pH was determined on a 1:1 soil/water slurry using a glass electrode (Peters et al., 2012). Soil organic matter was determined by loss on ignition (LOI) by placing the sample in a high temperature oven at 360°C for 2h (Combs and Nathan, 1998). Extractable Ca, Mg, K and S were measured using a Mechlich-III extractant solution (Mehlich, 1984) and analyzed with an inductively coupled plasma atomic emission spectrophotometer.

Data Analysis

The two gypsum application strategies were considered as independent and were analyzed separately as two different components of the same study. The data were subjected to analysis of variance using the PROC MIXED model in SAS v9.4 (SAS Institute, 2011) to determine the significance (at 5% level) of main (N rate) and sub-plot (either gypsum rate or gypsum rate frequency) factors and their respective interactions. Nitrogen and gypsum effects were assumed as fixed and block was considered a random effect, and year was considered as a repeated measure for the yield data.

Due to the unbalanced nature of this experimental design, separate analyses were conducted to evaluate for differences between ANOVAs that either had the zero N rate treatment included or not included in the model. Except for grain yield, no major differences were observed between the analyses with respect to the significance (p-values) of main and subplot factors and their respective interactions on other measured variables. However, full mean comparisons across all treatments were only possible when the 0 kg N ha^{-1} treatment was excluded in the analysis. Therefore, only the 84, 168, and 252 kg ha^{-1} N rates and either the three gypsum rate treatments or the three gypsum rate frequency treatments were included in the final model to allow for multiple mean comparisons for variables other than yield.

For yield data, when N rate effects were significant ($p \le 0.05$), mean comparisons were done at each N rate (including the 0 N) treatment across all gypsum treatments. Also, if the yield data was found to be significantly ($p \le 0.05$) different by year and site, ANOVA was run for each year and site separately. For tissue, grain and soil nutrient concentrations, when N rate was not significant ($p > 0.05$), means were calculated across all three plus N rates for each gypsum rate or gypsum rate frequency treatment. Mean comparisons between treatments were conducted using the adjusted Tukey's test in the LSMEANS routine in SAS at $p \le 0.05$, unless otherwise stated.

RESULTS AND DISCUSSION

Corn Grain Yield

Gypsum rate significantly increased ($p = 0.01$) corn grain yields at the Northwest site in only 2017 (Table 1). Relative to the no-gypsum treatment, mean grain yields were increased by 19.1 and 13.5% with the 1.1 and 2.2 Mg ha^{-1} gypsum rates, respectively, at the Northwest site in 2017. The gypsum frequency treatments showed an opposite trend at this site in 2017, where the one-time gypsum application rate of 4.4 Mg ha^{-1} resulted in significantly lower grain yields compared to the 1.1 Mg ha^{-1} annual and 2.2 Mg ha^{-1} bi-annual applications (Table 1). Mean yields were 10 and 8% lower for the one time 4-yr application, compared to the annual and biannual applications, respectively, which received the same amount of total gypsum applied over the same 4-yr period but at lower rates.

At the Wooster site, gypsum rate treatments had little effect on corn grain yields and did not lead to a significant yield increase in any of the 3 yr (Table 1). At the same site, in 2016, the 4-yr gypsum frequency treatment (4.4 Mg ha^{-1} of gypsum applied 4 yr prior to 2017) however, reduced yields compared to annual and bi-annual applications ($p < 0.05$). Mean yields were 9.5 and 15.5% lower for the 4-yr treatment compared to annual and bi-annual gypsum applications at this site in 2016.

Regardless of gypsum applications, corn was highly responsive to N application at both the study sites. Corn grain yield was significantly increased by fertilizer N rate in all years at both the Northwest and Wooster sites (Table 1). Mean yields ranged from 2.5 to 12.6 Mg ha^{-1} among N treatments at the Northwest site, and ranged from 1.9 to 9.8 Mg ha^{-1} at Wooster, across all years. No significant interaction between nitrogen and gypsum applications (either rate or frequency treatments) was found in any of the years at either site with the exception of the 2017 yr at the Wooster site, where a strong trend toward an interaction between N and gypsum frequency treatment was noted ($p = 0.06$; Table 1). It was found that yields tend to be reduced at lower N rates as the frequency of gypsum application increased.

Previously, Chen et al. (2008) reported a positive corn yield response to gypsum application when gypsum was applied at a 0.2 Mg ha^{-1} rate in two of the 4 yr (Chen et al., 2008). This yield response was however, attributed to the gypsum meeting

Table 1. Corn grain yields from 2014 to 2017 for N and gypsum rate and gypsum application frequency treatments at two study sites (mean ± SE).

	Northwest site				Wooster site†		
Variable	2014	2015	2016	2017	2015	2016	2017
Gypsum rate (GR, Mg ha^{-1})				Yield			
				Mg ha^{-1}			
0	8.4 ± 0.5	5.9 ± 0.6	9.1 ± 0.4	8.9 ± 0.7 b‡	6.0 ± 0.8	9.1 ± 0.4	7.6 ± 0.4
1.1	8.7 ± 0.5	6.7 ± 0.4	9.6 ± 0.2	10.6 ± 0.7 a	6.6 ± 1.2	8.4 ± 0.5	6.8 ± 1.1
2.2	8.3 ± 0.5	6.9 ± 0.7	9.3 ± 0.4	10.1 ± 0.9 a	6.2 ± 0.7	7.9 ± 0.5	7.8 ± 0.5
Nitrogen rate (N, kg ha^{-1})							
0	4.5 ± 0.5 c	2.5 ± 0.3 c	5.6 ± 0.4 b	3.5 ± 0.3 d	1.9 ± 0.3 b	3.8 ± 0.2 c	3.8 ± 0.6 b
84	8.3 ± 0.3 ab	4.9 ± 0.4 b	8.6 ± 0.2 a	7.5 ± 0.4 c	3.7 ± 0.1 b	7.6 ± 0.4 b	7.4 ± 0.3 a
168	7.5 ± 0.6 b	6.9 ± 0.5 a	9.6 ± 0.3 a	9.7 ± 0.5 b	6.2 ± 0.4 ab	8.9 ± 0.4 ab	6.5 ± 0.6 ab
252	9.8 ± 0.3 a	7.0 ± 0.7 a	9.8 ± 0.4 a	11.9 ± 0.6 a	8.9 ± 1.4 a	9.8 ± 0.5 a	9.2 ± 0.9 a
Source				P > F			
GR	0.79	0.12	0.46	0.01	0.78	0.12	0.98
N	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
N × GR	0.66	0.49	0.94	0.32	0.81	0.55	0.12
Gypsum Frequency (GF)				Yield			
				Mg ha^{-1}			
Annual	8.7 ± 0.5	6.6 ± 0.4	9.6 ± 0.2	10.2 ± 0.6 a	6.6 ± 1.2	8.4 ± 0.5 ab	7.6 ± 0.8
Bi–annual	8.8 ± 0.4	6.6 ± 0.7	9.5 ± 0.4	10 ± 0.8 a	6.9 ± 1.1	9.0 ± 0.4 a	7.9 ± 0.4
4–yr	7.9 ± 0.6	6.4 ± 0.7	8.9 ± 0.5	9.2 ± 0.9 b	5.6 ± 0.6	7.6 ± 0.3 b	7.5 ± 0.3
Nitrogen rate (N, kg ha^{-1})							
0	3.9 ± 0.4 c	2.5 ± 0.3 c	5.4 ± 0.2 c	3.4 ± 0.2 d	1.9 ± 0.3 b	3.8 ± 0.2 c	4.1 ± 0.6 b
84	8.1 ± 0.3 b	4.9 ± 0.5 b	8.6 ± 0.3 b	7.7 ± 0.4 c	3.8 ± 0.3 b	7.5 ± 0.4 b	7.2 ± 0.3 ab
168	8.1 ± 0.5 b	7.3 ± 0.4 a	9.8 ± 0.2 a	10.6 ± 0.3 b	7.0 ± 0.8 a	8.9 ± 0.4 a	7.0 ± 0.7 ab
252	10.1 ± 0.3 a	7.7 ± 0.5 a	10.3 ± 0.3 a	12.6 ± 0.3 a	9.6 ± 1.5 a	9.8 ± 0.4 a	9.5 ± 0.8 a
Source				P > F			
GF	0.24	0.65	0.06	0.01	0.22	<0.05	0.26
N	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
N × GF	0.91	0.19	0.26	0.28	0.84	0.13	0.06

† At the Wooster site, 2014 yield data are not available due to a crop failure.

‡ Means in a column followed by the same letter or no letter are not significantly different from each other at $P < 0.05$, adjusted Tukey's test.

a sulfur nutritional need by the crop. In the current study, the much higher gypsum application rates applied to the Wooster site did not cause a corn grain yield increase in any of the 3 yr. Evidently, the 1.1 to 4.4 Mg ha^{-1} rates of gypsum applied to this soil caused some sort of change in the soil's chemical, biological, or physical properties that resulted in no yield increase or even a very slight yield decrease. The exact reasons for this are not clearly understood at this time but it is possible that the Ca in the applied gypsum inhibited plant uptake of nutrients such as Mg and K. It is also possible that gypsum applied at higher rates improved soil permeability and subsequently the leaching potential. This might have leached some nutrients down the root zone and probably led to nutrient imbalances that negatively affected yield at the Wooster location. Nevertheless, inconsistent corn grain yield responses to gypsum application to silt loam soils have also been previously reported, even when applied at rates sufficient to meet the nutritional needs (O'Leary and Rehm, 1990; Kost et al., 2014).

Our results from the Northwest site are, however, closer to those reported by Sawyer et al. (2011) who found positive corn yield responses of gypsum applications to fine-textured soils in Northcentral and Northeastern Iowa. The positive yield response to gypsum application on finer textured soils has been generally attributed to S fertilization rather than the soil conditioning effect of gypsum (Sawyer et al., 2011). Sulfur fertilizer responses generally do not require the higher gypsum rates (e.g., 1.1 to 4.4 Mg ha^{-1}) investigated in this paper. Caires et al. (2016) reported an increase in root length to deeper layers on addition of gypsum at rates much higher than used in this current study, which enhanced N uptake and increased no-till corn yields in clay soils. This result was however, attributed to amelioration of subsoil aluminum toxicity (Al^{3+}) by gypsum applications (Caires et al., 2016). The positive response to gypsum observed in the current study was may be due to both meeting sulfur nutritional requirements of the corn crop and to changes in other soil properties as Al^{3+} toxicity is not common in soils of our study sites.

Also, our findings are contrary to studies that observed a significant N × S interaction and reported yield increases at lower N rates when S was applied at low rates (i.e., ~200 kg ha^{-1}) of gypsum (Chen et al., 2008; Steinke et al., 2015). Salvagiotti et al. (2009) also reported N × S synergism when S was applied at 30 kg ha^{-1}, which increased wheat grain yields by increasing the recovery efficiency of applied N. It is well known that N and sulfur have regulatory influence on each other (Fageria, 2001). Significant N × S interactions at lower N rates could be due to their effects on protein synthesis. Therefore, S and N applications can result in a positive yield response (Steinke et al., 2015). Sutradhar et al. (2017) however, reported that corn yields were increased by S application when N was applied at rates ≥203 kg N ha^{-1} but not at lower rates, in Minnesota soils. In our study,

Table 2. Corn ear-leaf (R1) nutrient concentrations for three gypsum rate and three gypsum application frequency treatments at two study sites (mean ± SE).

Site	Gypsum treatment	Ear–leaf tissue nutrients					
		N	P	K	Ca	Mg	S
		g kg^{-1}					
	Rate, Mg ha^{-1}						
Northwest	0	31.7 ± 1.47	4.07 ± 0.11	17.3 ± 0.47 b†	6.43 ± 0.16 b	1.25 ± 0.07	1.98 ± 0.07 c
	1.1	34.2 ± 1.77	4.11 ± 0.10	18.9 ± 0.49 a	6.54 ± 0.20 b	1.16 ± 0.02	3.05 ± 0.06 b
	2.2	34.3 ± 1.44	4.23 ± 0.10	19.1 ± 0.21 a	7.04 ± 0.10 a	1.15 ± 0.03	3.90 ± 0.12 a
Wooster	0	40.7 ± 0.86	4.24 ± 0.04	18.2 ± 0.39	7.75 ± 0.09	2.78 ± 0.08	2.73 ± 0.07 c
	1.1	41.6 ± 0.91	4.12 ± 0.09	17.9 ± 0.47	8.12 ± 0.33	2.73 ± 0.12	3.15 ± 0.19 b
	2.2	40.5 ± 0.76	4.14 ± 0.10	17.7 ± 0.46	8.34 ± 0.29	2.66 ± 0.12	3.75 ± 0.19 a
	Frequency						
Northwest	Annual	34.2 ± 1.76	4.11 ± 0.10	18.8 ± 0.49	6.48 ± 0.18	1.17 ± 0.01	3.04 ± 0.16 a
	Bi–annual	33.3 ± 2.29	4.10 ± 0.14	18.9 ± 0.26	6.35 ± 0.21	1.19 ± 0.02	2.56 ± 0.16 ab
	4–yr	31.8 ± 2.38	4.06 ± 0.12	18.3 ± 0.27	6.36 ± 0.21	1.17 ± 0.04	2.24 ± 0.11 b
Wooster	Annual	41.6 ± 0.90	4.12 ± 0.09	17.9 ± 0.5 a	8.12 ± 0.29	2.73 ± 0.11	3.13 ± 0.20
	Bi–annual	41.7 ± 0.74	4.31 ± 0.09	18.6 ± 0.4 a	8.05 ± 0.19	2.67 ± 0.11	3.21 ± 0.07
	4–yr	41.1 ± 0.63	4.14 ± 0.08	16.9 ± 0.33 b	8.33 ± 0.18	2.87 ± 0.07	2.85 ± 0.09

† Means in a column followed by the same letter or no letter are not significantly different from each other at $P < 0.05$, adjusted Tukey's test.

we did not observe any significant interaction effects of gypsum and N applications on corn yield response, either positive or negative, or at either lower or higher N rates at both study sites.

Kim et al. (2013) reported that organic matter content could also play an important role in providing a S response. They found that greatest S response was observed when soil organic matter (SOM) was <20 g kg^{-1}, low to moderate response between 20 to 40 g kg^{-1} of SOM and no response when SOM was >40 g kg^{-1} (Kim et al., 2013). Though Wooster soils in our study had organic matter content less than 20 g kg^{-1} (12.6 g kg^{-1}), corn response to gypsum applications was not found in any of the years. This could be due to the fact that soils at this site were not S deficient and gypsum application only supplemented the residual S supply of these soils. In contrast, the Northwest site had a significant corn yield response following 4 yr of continuous gypsum application (2017; Table 1) even though it had an organic matter content >20 g kg^{-1} (21.3 g kg^{-1}). As previously stated, it is not clear if this positive response was due to a S fertilization effect or a soil conditioning effect by gypsum.

Regardless of the rates at which gypsum was applied, corn yields generally showed a decreasing trend at both the study sites as the frequency of gypsum application was reduced (Table 1). This could mean that an initial single large application (4.4 Mg ha^{-1} applied one-time at the beginning of our 4-yr experiment) may have lead to yield reductions either due to reduced availability of nutrients or a chemical nutrient imbalance. Corn yields were not significantly different between bi-annual and annual gypsum applications at both the Northwest and Wooster sites. This suggests that where gypsum applications are beneficial, a bi-annual application could be equally effective as an annual application and therefore, the more economically viable option.

It should however, be noted that site-specific characteristics like the soil texture, could likely play a major role in deciding if gypsum applications are beneficial or not. For example, in this study, at the Northwest site, gypsum applications increased yields by 7 to 10% over the 4-yr period. At the same time, yields in fact, declined at the Wooster site over a 3-yr period (3–4% decrease). This likely suggests that gypsum application at the Wooster site may not be beneficial unless there are other soil and water quality benefits (e.g., reduced P loads in water leaving the field) that could overlook yield response to gypsum. So careful considerations must be made before adopting gypsum as a management practice as it may not be economically beneficial at all sites.

Ear-leaf Tissue Nutrient Concentrations

Gypsum rate, regardless of N rate, had a significant effect ($p < 0.01$) on ear-leaf tissue S concentrations at both sites (Table 2). Gypsum applied at 1.1 and 2.2 Mg ha^{-1} increased tissue S concentrations by 54 and 97% at the Northwest site, and 15 and 37% at the Wooster site, relative to the no gypsum control. Increase in tissue S concentrations following gypsum applications are expected as it is known to be an excellent source of S for plants (18.6% S; Watts and Dick, 2014). Increased ear-leaf S concentrations due to S fertilization were also reported in previous studies (Bullock and Goodroad, 1989; O'Leary and Rehm, 1990; Pagani and Echeverría, 2011; Kim et al., 2013; Michalovicz et al., 2014; Steinke et al., 2015).

The frequency of gypsum application had smaller influence on ear-leaf tissue S concentrations than the gypsum application rate. More frequent applications increased tissue S concentrations at the Northwest site, but not at the Wooster site (Table 2). But in general, as the frequency of gypsum application decreased, ear-leaf S concentrations also decreased, with the 4-yr application frequency resulting in the lowest S concentrations (Table 2). However, ear-leaf S concentrations were still within the ideal range (Vitosh et al., 1995) at both the study sites, regardless of gypsum application. Interestingly at the Northwest site in 2017, there was a significant yield increase with gypsum addition and a significant yield decrease with gypsum application rate frequency (Table 1). Tissue S concentrations were also found to follow the same trend as yields. This suggests that corn plants possibly encountered a S deficiency in 2017 at this site, even when the tissue S concentrations for the zero-rate (1.98 g kg^{-1}) and 4-yr gypsum frequency treatments (2.24 g kg^{-1}) were with the sufficiency range. This suggests that the current recommended critical ear-leaf tissue S concentration for corn ear-leaf (1.6 g kg^{-1}) may be insuffcent for adqequate corn S needs in fine-textured soils. Further studies are needed to confirm this finding as the present sufficiency range

Table 3. Corn grain nutrient concentrations for three gypsum rate and three gypsum application frequency treatments at two study sites (mean ± SE).

Site	Gypsum treatment	Grain nutrients					
		N	P	K	Ca	Mg	S
		g kg^{-1}					
	Rate, Mg ha^{-1}						
Northwest	0	10.7 ± 0.52	2.86 ± 0.09	3.34 ± 0.08	1.10 ± 0.01	0.91 ± 0.03	0.85 ± 0.03 b†
	1.1	10.2 ± 0.38	2.72 ± 0.09	3.36 ± 0.12	1.16 ± 0.09	0.88 ± 0.03	0.98 ± 0.04 a
	2.2	10.6 ± 0.49	2.71 ± 0.12	3.35 ± 0.10	1.10 ± 0.03	0.86 ± 0.02	1.00 ± 0.04 a
Wooster	0	13.9 ± 0.63	2.78 ± 0.05	3.94 ± 0.07	1.00 ± 0.01	1.07 ± 0.02	1.15 ± 0.02 b
	1.1	14.2 ± 0.37	2.69 ± 0.06	3.96 ± 0.05	1.03 ± 0.02	1.07 ± 0.01	1.23 ± 0.03 a
	2.2	13.7 ± 0.56	2.72 ± 0.08	3.91 ± 0.08	1.05 ± 0.02	1.05 ± 0.02	1.24 ± 0.03 a
	Frequency						
Northwest	Annual	10.2 ± 0.36	2.72 ± 0.09	3.49 ± 0.13	1.15 ± 0.07	0.90 ± 0.03	0.96 ± 0.04
	Bi–annual	9.95 ± 0.58	2.87 ± 0.09	3.50 ± 0.08	1.09 ± 0.01	0.93 ± 0.03	0.94 ± 0.03
	4–yr	11.0 ± 0.51	2.78 ± 0.11	3.42 ± 0.11	1.11 ± 0.01	0.88 ± 0.04	0.92 ± 0.04
Wooster	Annual	14.2 ± 0.38	2.69 ± 0.06	4.04 ± 0.06	1.02 ± 0.02	1.10 ± 0.01	1.20 ± 0.03
	Bi–annual	13.8 ± 0.56	2.74 ± 0.10	4.09 ± 0.11	1.02 ± 0.02	1.11 ± 0.03	1.19 ± 0.04
	4–yr	13.7 ± 0.70	2.63 ± 0.06	3.86 ± 0.06	1.05 ± 0.02	1.05 ± 0.02	1.21 ± 0.03

† Means in a column followed by the same letter or no letter are not significantly different from each other at $P < 0.05$, adjusted Tukey's test.

for corn ear-leaf S concentration given in the Tri-State Fertilizer Recommendations (Vitosh et al., 1995) is generalized across soils with no specific reference to different soil textures. Also, given the fact that a positive yield response was observed after 4 yr of continuous gypsum application at the Northwest site, it could be possible that gypsum produced a soil conditioning effect that improved soil physical properties. However, in the present study, no attempt was made to quantify soil physical properties.

Regardless of gypsum applications, tissue N concentrations increased significantly with N application, at both the Northwest and Wooster sites. Respective mean tissue N concentrations for the 0, 84, 168, and 252 kg N ha^{-1} treatments were 19.3, 27.3, 35.0, and 37.0 g kg^{-1} at the Northwest and 30.5, 41.3, 40.7, and 41.5 g kg^{-1} at the Wooster site. The mean R1 ear-eaf tissue N concentrations found in our study were within the sufficiency range reported by Vitosh et al. (1995) and Bryson et al. (2014), except at the Northwest site. Tissue N concentrations in 2017 for the 0 and 84 kg N ha^{-1} treatments (19.3 and 27.3 g kg^{-1}, respectively) at the Northwest site were below the critical level of 29 g kg^{-1} tissue N (Vitosh et al., 1995). This probably resulted in a very distinct yield response to N applications at the Northwest site in 2017 (Table 1). Nevertheless, increase in tissue N concentrations following N applications were also reported in previous studies (Kovács and Vyn, 2017; O'Leary and Rehm, 1990; Steinke et al., 2015; Sutradhar et al., 2017). These authors have also reported that tissue S concentrations were significantly increased by N application and concluded that N has a greater impact on S uptake when both N and S are applied simultaneously. In the current study, N applications did not have any significant effect on tissue S concentrations either with or without gypsum applications at both the study sites. This suggests that in our case, gypsum applications had a greater impact on tissue S concentrations rather than N applications, which is likely due to luxury consumption of S following gypsum applications. Sutradhar et al. (2017) reported rather interesting results in that S fertilization increased tissue S concentrations as well as ear-leaf tissue N concentrations but did not find any interaction between N and S. Changes in tissue N concentrations due to gypsum (i.e., S) applications were however, not observed in the current study.

Ear-leaf tissue Ca concentrations were increased in general as the gypsum application rate increased. But the increase was significant only at the Northwest site (Table 2). Higher Ca concentrations in the ear-leaf tissue can be attributed to increased Ca availability in the soil for plant uptake following gypsum additions. Whereas, gypsum rate frequency did not significantly affect tissue Ca concentrations at either of the study sites.

Consistent with tissue Ca, tissue K concentrations were also significantly increased by gypsum applications at the Northwest site, but were not affected at the Wooster site (Table 2). Increased K concentrations in ear-leaf tissue, following gypsum addition, has been seen in other studies (unpublished data), but this is not a consistent observation reported in literature. The reason for the increased K concentrations in ear-leaf samples can be attributed to Ca displacing K and moving it into the soil solution where it is more available for plant uptake. Interestingly, ear-leaf tissue K decreased as the frequency of gypsum application decreased at the Wooster site, with the 4-yr frequency treatment resulting in the lowest tissue K levels (Table 2). Reason for this result is unclear but could be from the competitive mass flow of K^+ off exchange sites from the large gypsum application (4.4 Mg ha^{-1}) 4 yr prior.

Grain Nutrient Concentrations

Gypsum addition significantly increased grain S concentrations compared to no gypsum application at both the Northwest and Wooster ($p < 0.1$) sites (Table 3). Grain S concentrations were increased on average by 0.2 and 0.1 g kg^{-1} at the Northwest and Wooster sites, respectively, when gypsum was applied at either the 1.1 or 2.2 Mg ha^{-1} application rates. Grain S concentrations were also found to be increased on average by 0.1 g kg^{-1} with S fertilization in the Sutradhar et al. (2017) study. Chen et al. (2008) also reported that S fertilization through gypsum additions increased grain S concentrations when corn was grown in a Wooster silt loam soil. In addition, significantly higher grain S concentrations were observed after S fertilization of both medium and fine-textured soils in Minnesota (Kim et al., 2013) and of coastal plain soils (Rabuffetti and Kamprath, 1977). Gypsum application frequency however, did not affect grain

Table 4. Soil nutrient concentrations for three gypsum rate and three gypsum application frequency treatments at two study sites (mean ± SE).

Site	Gypsum treatment	Soil nutrient concentrations					
		Soil pH	Ca	Mg	K	S	P
			mg kg^{-1}				
	Rate, Mg ha^{-1}						
Northwest	0	6.28 ± 0.06	2297 ± 50 b†	345 ± 6.8 a	166 ± 6.1	14.4 ± 1.7 c	26.6 ± 1.18
	1.1	6.27 ± 0.06	2377 ± 46 ab	311 ± 6.8 b	161 ± 5.7	43.1 ± 7.2 b	24.1 ± 1.34
	2.2	6.16 ± 0.09	2596 ± 99 a	303 ± 15 b	166 ± 9.3	113 ± 9.8 a	25.6 ± 1.33
Wooster	0	6.47 ± 0.18	1116 ± 41 b	201 ± 9.5	54 ± 1.9	11.1 ± 0.6 c	19.4 ± 0.77
	1.1	6.43 ± 0.17	1192 ± 48 ab	193 ± 7.5	53 ± 1.5	40.2 ± 5.3 b	18.5 ± 1.21
	2.2	6.39 ± 0.15	1261 ± 49 a	189 ± 6.6	52 ± 1.9	70.1 ± 8.8 a	18.0 ± 0.96
	Frequency						
Northwest	Annual	6.27 ± 0.06	2377 ± 46	311 ± 6.8	161 ± 5.7	43.1 ± 7.2 a	24.1 ± 1.34
	Bi–Annual	6.32 ± 0.06	2390 ± 44	314 ± 8.1	170 ± 6.1	23.6 ± 2.2 b	25.4 ± 0.94
	4–yr	6.41 ± 0.05	2379 ± 58	312 ± 9.9	160 ± 7.8	14.8 ± 0.5 c	23.7 ± 1.11
Wooster	Annual	6.43 ± 0.17	1192 ± 48	193 ± 7.5	53 ± 1.5	40.2 ± 5.3 a	18.4 ± 1.21
	Bi–Annual	6.27 ± 0.17	1158 ± 44	193 ± 10.7	53 ± 1.7	26.8 ± 2.7 b	19.7 ± 0.82
	4–yr	6.44 ± 0.14	1201 ± 30	207 ± 7.6	54 ± 1.8	16.0 ± 1.2 c	18.0 ± 1.19

† Means in a column followed by the same letter or no letter are not significantly different from each other at $P < 0.05$, adjusted Tukey's test.

S concentrations. This suggests that the frequency of gypsum application does not matter as long as sufficient levels of S are maintained in the soil to meet the plant demand.

Regardless of gypsum application, similar to tissue N, grain N concentrations were also increased by N fertilizer applications at both the Northwest and Wooster sites. Respective mean grain N concentrations for the 0, 84, 168, and 252 kg N ha^{-1} treatments were 9.4, 9.3, 10.4 and 11.7 g kg^{-1} at the Northwest and 10.7, 12.9, 14.6 and 14.1 g kg^{-1} at the Wooster site.These results are in conjunction with those reported by Chen et al. (2008) and Sutradhar et al. (2017) where N fertilizer applications significantly increased grain N concentrations, irrespective of S fertilization.

The gypsum rate or the frequency of application did not have any significant effect on any of the other grain nutrient concentrations (Table 3). Also, there was no significant interactions between nitrogen and gypsum applications found in this study with respect to grain nutrient concentrations.

Soil Nutrient Concentrations

Regardless of N application, gypsum addition significantly increased Mechlich III extractable soil S levels at both the Northwest and Wooster sites (Table 4). At the Northwest site, gypsum applied at 1.1 and 2.2 Mg ha^{-1} rates increased S concentrations by 3- and 8-fold, respectively, relative to the control. Similarly, at the Wooster site, gypsum application at 1.1 Mg ha^{-1} and 2.2 Mg ha^{-1} rates increased soil S concentrations by 4- and 7-fold respectively, compared to the control. Gypsum application has been found to increase soil S levels, even at much lower rates than were applied in this study (Chen et al., 2008; Kost et al., 2014). On the other hand, reduced frequency of gypsum application significantly decreased extractable soil S concentrations at both the sites. A 4-yr gypsum application frequency resulted in soil S levels dropping to levels those observed in control soils, which did not receive any gypsum (Table 4). At the Northwest site, soil S concentrations were almost equal for both the zero-rate gypsum and the 4-yr gypsum frequency treatments (14.4 vs. 14.8 mg kg^{-1}, respectively). Similarly, soil S levels at the Wooster site were 11.1 and 16.0 mg kg^{-1} for the zero-rate gypsum and 4-yr frequency treatment, respectively. This suggests that single large applications of gypsum lose the benefit of sulfur fertilization within a few years, likely because of S losses, primarily through leaching.

Moreover, at the Northwest site, soil S levels observed for the 4-yr application frequency treatment and the zero-rate gypsum treatment (Table 4), were likely insufficient and may have caused S deficiency that resulted in significantly lower yields in 2017 (Table 1). Bi-annual gypsum applications however, resulted in S concentrations that were well above the levels observed in soils with no added gypsum (Table 4). This indicates that bi-annual applications of gypsum could maintain more than enough SO_4–S levels in the soil. Annual applications, though result in higher soil S concentrations, would offer no performance advantage. This is evident from both the grain yield and ear-leaf tissue S data (Tables 1 and 2), where differences in tissue S concentrations and grain yields were not significantly different among annual and bi-annual gypsum applications. From this study, it therefore appears that bi-annual applications are more economical than annual applications in terms of transportation and application costs.

Extractable Ca levels were also significantly increased with increasing gypsum application rates at both the study sites. Previous studies have also reported increased extractable soil Ca levels after gypsum applications (Shamshuddin et al., 1991; Kost et al., 2014; Michalovicz et al., 2014). Gypsum is known to readily impact extractable or available Ca due to its relatively higher solubility compared to other Ca sources (Shainberg et al., 1989; Watts and Dick, 2014). However, given the amount of gypsum applied, it was surprising that the magnitude of increase was less than expected. It could be possible that gypsum might have solubilized completely and Ca might have leached deeper into the soil profile. On the other hand, the frequency of gypsum application did not significantly affect the soil Ca levels at both the sites.

Increasing gypsum application rate significantly decreased extractable soil Mg concentrations at only the Northwest site. Excess Ca after gypsum additions likely resulted in the removal of Mg from soil exchange sites into soil solution, followed by its leaching out of the soil profile (Syed-Omar and Sumner, 1991). Therefore, soil Mg concentrations decreased with gypsum additions at the Northwest site. The frequency of gypsum application on the otherhand did not significantly affect the soil extractable Mg levels at both the sites.

Either the gypsum rate or the frequency of application did not have any significant effects on the P and K nutrient status. Soil pH was also not affected by either the gypsum rate or the frequency of application. Gypsum is not expected to influence soil pH as it is a neutral salt. Nitrogen rate, regardless of gypsum applied, did not have any statistically significant effect on soil pH and any of the soil nutrient concentrations.

Soil organic matter contents ranged from 20.6 to 22.2 g kg^{-1} at the Northwest location and 12.5 to 14.4 g kg^{-1} at the Wooster location. Neither of the gypsum and nitrogen applications caused significant changes in the soil organic matter content at either of the study sites.

CONCLUSIONS

Application of gypsum at higher agronomic rates did not produce any yield benefit in most years at the two study sites. Lack of N and gypsum interaction at both sites indicates that N and gypsum were independent of each other and the efficiency of N use was not enhanced even with high rates of gypsum application. The results from this study therefore reiterate that the ability of gypsum applications to produce a positive corn yield response and improved N use, even when applied at higher rates, can be very inconsistent and depends greatly on site-specific soil characteristics. Even though the ear-leaf tissue S concentrations were within the sufficiency range, yield differences among gypsum treaments were observed at the Northwest site in 2017. This indicates that there was possibly a S deficiency at this site and that the critical tissue S concentration for corn grown in fine-textured soils may be higher than what is currently recommended.

Gypsum applied at 2.2 Mg ha^{-1} annual rate did not offer any performance advantages or disadvantages over the 1.1 Mg ha^{-1} annual application rate. Corn receiving bi-annual applications of gypsum performed similar to annual applications in grain yield as well as tissue, grain and soil nutrient levels. Lower soil S levels, closer to control soils, observed in the 4-yr gypsum treatment indicates that single large applications can lead to reduced S availability overtime.

Our data suggests that applying gypsum at an annual application rate of 1.1 Mg ha^{-1} or applying 2.2 Mg ha^{-1} every 2 yr (bi-annual) optimizes any potential benefits associated with gypsum addition. Considering that a large percentage of the cost of gypsum is in the transportation and application, the bi-annual application would likely be a more economical option.

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