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Impact of Flue Gas Desulfurization Gypsum Applications to Corn-Soybean Plots on Surface Runoff Water Quality

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Abstract Row crop agriculture systems are a significant contributor to non-point source nutrient loading into water bodies. One approach to reduce phosphorus (P) losses through surface runoff is applying flue gas desulfurization (FGD) gypsum as a soil amendment. This research was conducted to examine the effects of different rates of FGD gypsum application to corn (*Zea mays* L.)–soybean (*Glycine max*) plots on water quality parameters including dissolved reactive phosphate (DRP), total phosphorus (TP), and total suspended solids (TSS). The study was conducted on a high P level (>30 mg P kg^{-1}) soil in a completely randomized design with four treatments each replicated three times. The four treatments were no FGD gypsum (control), FGD gypsum at a rate of 2.2 Mg ha^{-1}, FGD gypsum at 4.5 Mg ha^{-1}, and FGD gypsum at 13.5 Mg ha^{-1}. Gypsum applications were effective in reducing P loads in surface runoff water, with a significant ($P < 0.1$) reduction in DRP and TP from all the treatments compared to the control during the initial post-gypsum application period (December 2018–May 2019). Results suggest application rates of 4.5 Mg ha^{-1} and 13.5 Mg ha^{-1} were most suitable to reduce P loads in surface runoff water from Hosmer silt loam soil with high soil test P (STP) prior to P fertilizer application. However, following P fertilizer application (May 2019–January 2020), gypsum was not effective in reducing P in surface runoff. Overall, FGD gypsum appeared to be an effective phosphorus abatement tool for southern Illinois soils to improve water quality. Though, how long it remains effective

Core Ideas

- Flue gas desulfurization (FGD) gypsum applications reduced phosphorus (P) loads in surface runoff.
- FGD gypsum at a rate of 2.2 Mg ha^{-1} was an effective P abatement tool in Hosmer silt loam soils.
- P fertilization reduced the P abatement effectiveness of FGD gypsum in subsequent runoff events.
- Heavy metals were not increased in soil or surface runoff following FGD gypsum application.

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appears to be in question given our results in the post P fertilization period.

Keywords Sediment loss · FGD gypsum · Soil properties · Dissolved reactive phosphorus

Abbreviations

P	Phosphorus
FGD	Flue gas desulfurization
DRP	Dissolved reactive phosphorus
TP	Total phosphorus
TSS	Total suspended solids
STP	Soil test P

1 Introduction

The National Water Quality Inventory in 2017 identified agricultural runoff as a leading cause of impairment of water bodies in the United States (U.S.) (USEPA, 2017). A recent assessment of water quality trends in U.S. rivers has identified P as a primary cause of degradation of water bodies (Shoda et al., 2019). There has been a 75% increase in net P storage in terrestrial and aquatic ecosystems as indicated by the global P budget, mainly due to P fertilization of agricultural soils (Daryanto et al., 2017; Bennett et al., 2001; Zhou et al., 2017).

The amount of P delivered to water bodies is influenced by various factors, including the soil physical and chemical properties, plant-available P (soil test P, STP), the P source, rate and method of application, and transport factors (Sharpley et al., 1994). Phosphorus is strongly absorbed to soil particles, and consequently, total P losses from agriculture tend to be associated with surface runoff and soil erosion (Sharpley et al., 1998). There is increasing evidence that dissolved reactive phosphate levels in surface runoff can be significant when soil P levels are high (Singh et al. 2020; Randall & Mulla, 2001). Dissolved reactive phosphate (DRP) is transported in surface runoff and soil water, while particulate P transported is bound to sediment particles that are either redeposited within the field or exported from the field (Osmond et al., 2019). The recent increase in total phosphorus (TP) reported in the Illinois River was attributed to DRP rather than particulate phosphorus (PP), which may be associated with point sources as well as leaching from cropland and surface runoff of unincorporated P fertilizers (David et al., 2013). Agriculture and point sources contributed about 48% of the total phosphorus (TP) load exported by Illinois rivers, and 4% was from urban runoff (David et al., 2013).

Phosphorus is a major macronutrient required by plants and its application is necessary to obtain optimum agronomic production (Haegele et al., 2014). Dissolved P in surface runoff is strongly correlated to soil P content in surface soil horizons (Vadas et al., 2005). Sharpley et al. (2003) found that DRP in surface runoff was a linear function of STP inputs. Therefore, reducing STP would lead to a proportional reduction in DRP loss. Thus, the export of P in surface runoff is directly related to field management practices, making it essential to improve long-term P management in agriculture fields (Buda et al., 2009). To reduce P losses, different conservation practices have been prescribed such as improved P application techniques, in-field management practices such as cover crops, and edge of field practices such as riparian buffers that can attenuate P in surface runoff and soil water before it enters streams (Singh et al., 2020). The adoption of practices that retain more P in the field to be used by crops is economically as well as environmentally beneficial.

A variety of conservation practices are available to reduce P losses from agriculture fields (Singh et al., 2020, Dodd & Sharpley, 2016, Sharpley & Tunney, 2000). A recent concept proposed by USDA-NRCS (2019b) is "avoid, control, and trap," which signifies that control strategies should be adopted to address multiple facets by reducing P loss pathways, runoff, and leaching. Soil amendment with flue gas desulfurization (FGD) gypsum is one such potential control strategy for P (Wang & Yang, 2018; Endale et al., 2014). Flue gas desulfurization gypsum is a by-product of coal-burning electric power plants produced during the flue gas scrubbing process that removes sulfur from the flue gas per the regulations of the Clean Air Act. During the wet scrubbing process, a lime or limestone reagent is injected into the flue gas path to capture SO_2 as $CaSO_3$, which is then converted to $CaSO_4$ through forced air oxidation (Watts & Dick, 2014). Continuous increases in FGD production combined with a reduction in landfill spaces available created the need to find alternative uses, mainly in wallboard and agriculture. Due to its high purity, FGD gypsum is a quality source of gypsum and has been evaluated and approved for use as a soil amendment (Torbert & Watts, 2014; USDA-NRCS, 2015). Recent studies have shown reductions in P concentration in surface runoff following FGD gypsum application as it decreases P solubility (Watts & Torbert, 2009; Jaakkola et al., 2012). The FGD gypsum can also increase soil aggregate stability, therefore reducing surface runoff volume (Norton & Dontsova, 1998; Zhang et al., 1998; Favaretto et al., 2006). After FGD gypsum is applied to soils, it dissociates into Ca^{2+} and SO_4^{2-}. This Ca can readily combine with P resulting in the formation of an insoluble precipitate, Ca phosphate, which is relatively stable in soil (Elrashidi et al., 2010; Moore & Miller, 1994).

Gypsum application to soils has multiple positive impacts on soil physical and chemical properties, which positively affect crop production and environmental quality (Clark et al., 2001; Truman et al., 2010). However, along with the benefits of the FGD gypsum application, some environmental consequences may also result from its application. These consequences include the potential addition of heavy metals and non-toxic elements in aquatic as well as terrestrial ecosystems (Clark et al., 2001). Limited research has been conducted to understand the effect of FGD gypsum on water quality and its environmental consequences. Indeed, numerous laboratory studies have shown that FGD gypsum decreases P solubility, but there is a paucity of in-field research studies. The application of gypsum has already been added to conservation practices in the Midwestern states of Indiana and Ohio, but not in Illinois. The objective of this study was to evaluate the impacts of FGD gypsum application rate on surface runoff water quality, as measured by DRP, TP, suspended solids, and heavy metals from a field under a corn-soybean rotation in southern Illinois.

2 Material and Methods

2.1 Site Location and Experimental Design

The research site was located at Southern Illinois University Farms in Carbondale, IL, in a field containing a Hosmer silt loam (Fine-silty, mixed active, mesic Oxtaquic Fragiudalfs) soil series. Hosmer silt loams are characterized as moderately well-drained soil with a 2–5% slope and a very high to high runoff class with a depth to restricting layer between 60 and 90 cm and moderately permeable. The depth of the water table at the study area ranged from 0.45 to 1 m. The field was in a corn-soybean rotation, with no recent manure application and no-tillage with high soil test P level (> 67 kg ha^{-1}). The mean 10-year (2008–2018) annual precipitation for this research site is 1184 mm and the mean annual temperature is 14°C. The total precipitation at the study location from December 1, 2018, to January 31, 2020, was 1656.8 mm.

In spring 2018, twelve surface runoff plots 4.8 m^2 in area were installed in the soybean field using aluminum sheet metal edging pounded into the soil to a depth of 10 to 12 cm. Each runoff plot received one of the four FGD gypsum treatments 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, 13.5 Mg ha^{-1}, and control. The experimental design was a completely randomized design with three replications of each treatment (Fig. 1). Under NRCS recommendations, when the STP level is greater than twice the optimum level (45 kg ha^{-1}),

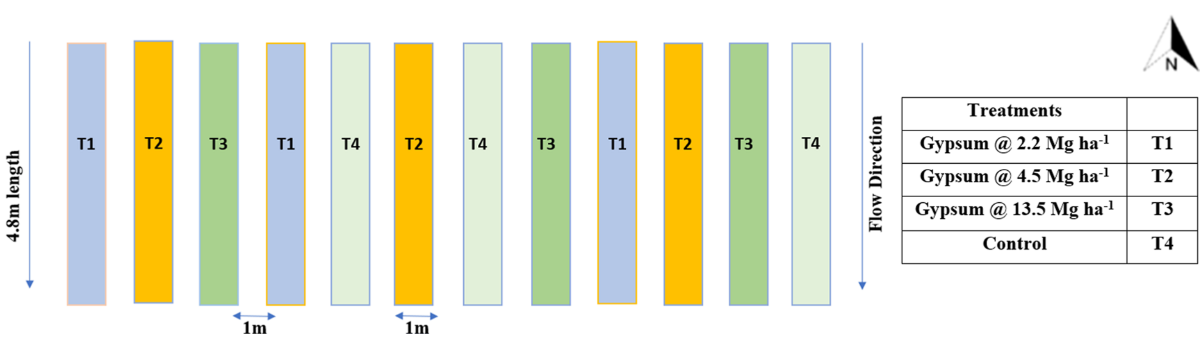

Fig. 1 Complete randomized treatment design for surface runoff study at the SIU Carbondale University Farms

2.2 Mg ha^{-1} of gypsum should be broadcasted to reduce the DRP level. The amount of gypsum can be increased up to 4.5 Mg ha^{-1} if water quality is a concern in the watershed, but beyond this rate, the anticipated decrease in P level is not proportional to gypsum cost (USDA-NRCS, 2015). Runoff from each plot flows through a 14-m-long PVC pipe and was collected in a 208 L high-density polyethylene (HDPE) plastic barrel laid on its side. Barrels were acid washed before installation using 1% hydrochloric acid (HCl) solution. A side port was constructed on the top of each barrel to pump out the water samples.

The FGD gypsum was purchased from Boral Resources at the Prairie State Energy Campus, Marissa, IL. Prior to field application of FGD gypsum, an independent sample of the Prairie State FGD gypsum was collected and analyzed for Ca, S, and heavy metal analysis by Brookside Laboratories (New Bremen, Ohio). Lab results reported FGD gypsum having 26% Ca and 18% S and had non-detectable or low levels of heavy metals. Following soybean harvest in the Fall 2018, gypsum treatments were hand weighed and hand-applied using a broadcast spreader. The plots were left fallow in winter 2018 and 2019. Soybean was planted in spring 2018 and corn was planted in spring 2019. In spring 2019 corn (DKC5645) was hand planted on May 5, 2019, at 75-cm row spacing and seeded every 15 cm following NPK fertilizer application. In 2019, urea, diammonium phosphate, and potassium chloride fertilizers were hand broadcasted at a rate of 159 kg N ha^{-1}, 22.5 kg P ha^{-1}, and 55 kg K ha^{-1} on May 3, 2019.

2.2 Sampling and Analysis

Runoff water samples were collected from December 15, 2018, to January 10, 2020. After each significant rainfall (≥12.5 mm), a 0.5 L water sample was collected from each barrel after stirring properly to suspend the sediments in the sample. The volume of water collected in the barrel was measured by a flow meter attached to a pump that emptied each barrel after a runoff event and samples were transported to the Southern Illinois University Forestry water quality lab.

In the laboratory, a water sample was subdivided into two bottles: a filtered and an unfiltered sample. The sample was filtered through a 0.45-μm nominal pore filter, stored in a refrigerator, and was used for dissolved reactive phosphorus (DRP) and sulfate-S (SO_4-S) analysis. Unfiltered samples were analyzed for water pH and electrical conductivity (EC) using a Fisher Scientific AT 20 pH/Conductivity meter (Fisher Scientific, Indiana, PA). The unfiltered sample was also used to determine total suspended solids (TSS) and total phosphorus (TP) and stored in the refrigerator below 4°C. Using 50 to 100 mL of unfiltered sample, TSS was determined using vacuum filtration following the 2540 D Total Suspended Solids method (Eaton et al., 2005). A Dionex ICS 2000 Ion Chromatograph (Dionex, 2005) was used for determining SO_4-S concentration in water (2000isp, Dionex, Thermo Fisher Scientific, Waltham, MA, USA). The DRP and TP levels in surface runoff water were analyzed using PerkinElmer Lambda 25 UV/Vis spectrophotometer (PerkinElmer Inc, Waltham, MA) using the ascorbic acid method (4500PE) (Alleman et al., 1995). Dissolved reactive phosphorus, TP, and SO_4-S loads were calculated using their concentration in the runoff multiplied by the volume of water collected in the barrel.

In total, 31 surface runoff events were collected over the post-treatment period of the study from December 2018 to January 2020. For performing statistical analysis, runoff events were divided into two-time frames based on the date of fertilizer application and were named as initial post-gypsum application period and post-fertilizer application period. The December 2018 to May 2019 period was referred to as the initial post-gypsum application period and the post-fertilizer application period included May 2019 following fertilization to January 2020. Based on the runoff plot area and rainfall amount, the calculation for runoff was obtained as maximum runoff to occur if no infiltration occurs and a minimum amount of runoff was expected if 85% of the precipitation infiltrated. The runoff volume values outlying these upper and lower limits were not included in the data analysis. A total of 15 events were analyzed during the initial-post gypsum application period and 16 events after P fertilizer application.

Nutrient loads of DRP, TP, TSS, and SO_4-S were determined by multiplying nutrient concentrations by corresponding flow volumes. Soil and runoff water samples were collected following gypsum application to identify any addition of heavy metals in soil and water. Surface runoff samples from the first event following treatment application were collected on

December 15, 2018, and soil samples were collected to a depth of 0–15 cm. The collected soil and water samples were analyzed for heavy metal analysis for trace elements including As, Cu, Cd, Hg, Mo, Ni, Pb, Se, Zn, and Cr by Carbondale Central Laboratory, Carbondale, IL (Martin et al., 1994).

2.3 Soil and Plant Sampling and Analysis

Soil samples were collected for soil quality analysis including Mehlich 3 extractable soil fertility, Bray I-P, and soil physical properties including bulk density, soil infiltration, and wet soil aggregate stability. Two samples were collected from the area adjacent to each runoff area using a push probe (JMC soil core sampler) at 0–5 cm and 5–15 cm soil depth for standard fertility analysis to minimize the disturbance of soil sampling inside the runoff plots. The area adjacent to each flume received the same gypsum treatment as the treatment applied in the flume. Soil samples were collected in the fall after crop harvesting before as well as after treatment application. The collected soil samples were air-dried, ground, and shipped to the Brookside Laboratories (New Bremen, Ohio) for standard fertility analysis including Bray-I P. Infiltration data were collected in the field during fall of 2018 and fall 2019 from the area adjacent to the runoff plot using a single-ring Cornell Sprinkler Infiltrometer (Cornell University, Ithaca, NY) (Infiltrometer, 1901) (Ogden et al., 1997). For measuring soil physical properties (infiltration rate, bulk density, and wet soil aggregate stability), one sample was collected at 0–15-cm depth from the area adjacent to each runoff plot. The collected soil samples for aggregate stability were analyzed according to Kemper and Rosenau (1986), at the SIU Department of Forestry's Soils Laboratory. Bulk density samples were analyzed according to Grossman and Reinsch (2002). Plant biomass samples were collected by manually harvesting 1 m^2 from each runoff area before grain harvesting. The collected samples were weighed and dried in oven at 60°C until constant weight per sample was recorded. Dried biomass samples were chopped and further ground to a fine powder (1-mm sieve). Dried and ground plant biomass samples were shipped to Brookside Laboratories (New Breman, OH) and analyzed using nitric acid and hydrogen peroxide digestion in a CEM Mars Express microwave system. The digested samples were then analyzed on a Thermo 6500 Dou ICP for S, P, Ca, Mg, K, Fe, Mn, B, Cu, Zn, and Al and scaled to g kg^{-1}.

2.4 Statistical Analysis

Runoff data collected during the study period were analyzed using a generalized mixed model procedure with the Glimmix statement in SAS Statistical software v9.4 (SAS Institute, Cary, NC). The soil and plant biomass samples were analyzed using the ANOVA procedure in SAS. Before analysis, all variables were tested for normality using the univariate procedure. Based on the Shapiro-Wilk test and Kolmogorov-Smirnov test used for determining normality of data, SO_4-S and runoff data were log-transformed for final analysis. For reporting data in tables, non-transformed data were displayed. All the variables including runoff volume, DRP, TP, and TSS loads and concentrations were analyzed by one factor: treatment with four levels. Additionally, cumulative loads were calculated for the DRP, TP, S, and runoff volume for a data collection period of 1 year with the Glimmix procedure. Soil samples were analyzed separately for each sampling depth (0–5 cm and 5–15 cm). Treatments were treated as a fixed factor and replication was treated as a random factor. For comparison of means, T-grouping and least-square means were calculated at alpha = 0.1 due to variability in data.

3 Results and Discussion

3.1 Surface Runoff

Surface runoff volume was significantly (P <0.0366) reduced in all FGD gypsum treatments 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, and 13.5 Mg ha^{-1} by 28%, 33%, and 43%, respectively, compared to control during the initial post-gypsum period (Table 1). However, during the post-fertilization period, no significant (P = 0.8800) effect on runoff volume was observed. Additionally, no significant reduction of cumulative runoff (P = 0.3951) was observed during the study period (December 2018–January 2020) (Fig. 2). The reduction in discharge was attributed to gypsum application reducing clay dispersion and sealing of the soil surface (Zhang et al., 1998). Zhang et al. (1998) observed elevated ionic strength in the soil solution

Table 1 Comparison of water runoff, mean total suspended solids (TSS), sulfur (S), total phosphorus (TP), and dissolved reactive phosphorus (DRP) load and concentration values during initial post-gypsum application period and post-fertilization period. Means within a column followed by the same letter are not statistically different (α = 0.1)

Treatment	Runoff	Concentration				Load			
		TSS	S	TP	DRP	TSS	S	TP	DRP
	L	mg L^{-1}				kg ha^{-1}			
Initial post-gypsum application period									
Control	125 a	166	1.8 d	1.7 a	1.1 a	49.3	0.5 d	0.5 a	0.31 a
2.2 Mg ha^{-1}	90 ab	237	12.2 a	1.5 ab	0.9 ab	36.9	3.4 a	0.3 b	0.20 b
4.5 Mg ha^{-1}	84 b	249	40.7 b	1.4 b	0.8 b	36.3	9.4 b	0.3 b	0.17 b
13.5 Mg ha^{-1}	72 b	238	78.4 c	1.4 b	0.9 b	28	13.8 c	0.2 b	0.15 b
p-value	0.0366	0.3426	<0.0001	0.0773	0.0312	0.4527	<0.0001	0.0073	0.0032
Post-fertilization period									
Control	71	266	0.8 d	2.6	1.8	37.8	0.1 d	0.4	0.26
2.2 Mg ha^{-1}	75	256	2 a	2.3	1.8	28.1	0.2 a	0.4	0.31
4.5 Mg ha^{-1}	66	196	4.7 b	2.3	1.9	22.1	0.4 b	0.3	0.27
13.5 Mg ha^{-1}	67	198	19.8 c	2.1	1.7	22.3	1.8 c	0.3	0.27
p-value	0.8800	0.3720	<0.0001	0.2414	0.4956	0.8990	<0.0001	0.5503	0.8202

and increased the Ca^{2+} fraction on soil exchange sites following gypsum application. However, soil infiltration rates (P = 0.5674) and Ca^{2+} concentrations (P = 0.3283) at 0–5-cm soil depth were similar among treatments, whereas, increased Ca^{2+} in 13.5 Mg ha^{-1} gypsum treatment was observed at 5–15-cm soil depth. In addition, high percentage of Mg on exchange sites in Midwestern soils can deteriorate soil structural properties and lower infiltration rates compared to soils high in Ca (Dontsova and Norton 2002). Therefore, reduced runoff volume in gypsum treated plots can be attributed to improved soil structural properties due to reduced Mg. In Georgia, Truman et al. (2010) reported that FGD gypsum applied at 9 Mg ha^{-1} reduced runoff loss by 40% on loamy sand soils when compared to control. The pH value for surface runoff water was within the range of 6.0–7.5 during the data collection period.

3.2 Phosphorus Concentration and Load

The DRP and TP concentration and load values in surface runoff were significantly lower (P < 0.1) in gypsum treatments compared to control for the initial post-gypsum application period (Table 1). Application of gypsum before fertilizer application (December 2018–May 2019) resulted in a reduction in DRP load (P = 0.0032) by 36%, 45%, and 52% compared to control in FGD gypsum treatments at 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, and 13.5 Mg ha^{-1}, respectively. Similarly, TP load (P = 0.0073) was reduced by 33%, 45%, and 55% compared to control in FGD gypsum treatments at 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, and 13.5 Mg ha^{-1}, respectively, during post-fertilization period. Cumulative TP (P = 0.0745) loads were reduced by 48% and 42% with 13.5 Mg ha^{-1} compared to control during the study period (Fig. 2). Gypsum treatments 4.5 Mg ha^{-1} and 13.5 Mg ha^{-1} reduced cumulative DRP (P = 0.0343) by 25% and 37% compared to control plots. These reductions in P loads were likely a result of gypsum converting soluble P in the soil to a less soluble calcium phosphate precipitate.

During the post-fertilization period (May 2019–January 2020), there was no significant effect of gypsum treatments on DRP (P > 0.1), and TP (P > 0.1) concentration as well as loads. This may have been due to the applied P in the fertilizer overwhelming any remaining P abatement effectiveness of the previous gypsum applications in the plots. The research plots were under no-tillage from the last 7 years (2012–2018) which might have resulted in the stratification of P in the topsoil layer leading to relatively high DRP losses, as evidenced by the control plot data. Some studies have reported no-tillage system can increase DRP concentrations and loads especially during wet years (Daryanto et al., 2017; Sharpley et al., 1994). When P is surface applied as a fertilizer, it leads to the accumulation of soluble P at the top of the soil surface and also increases the concentration of extractable P

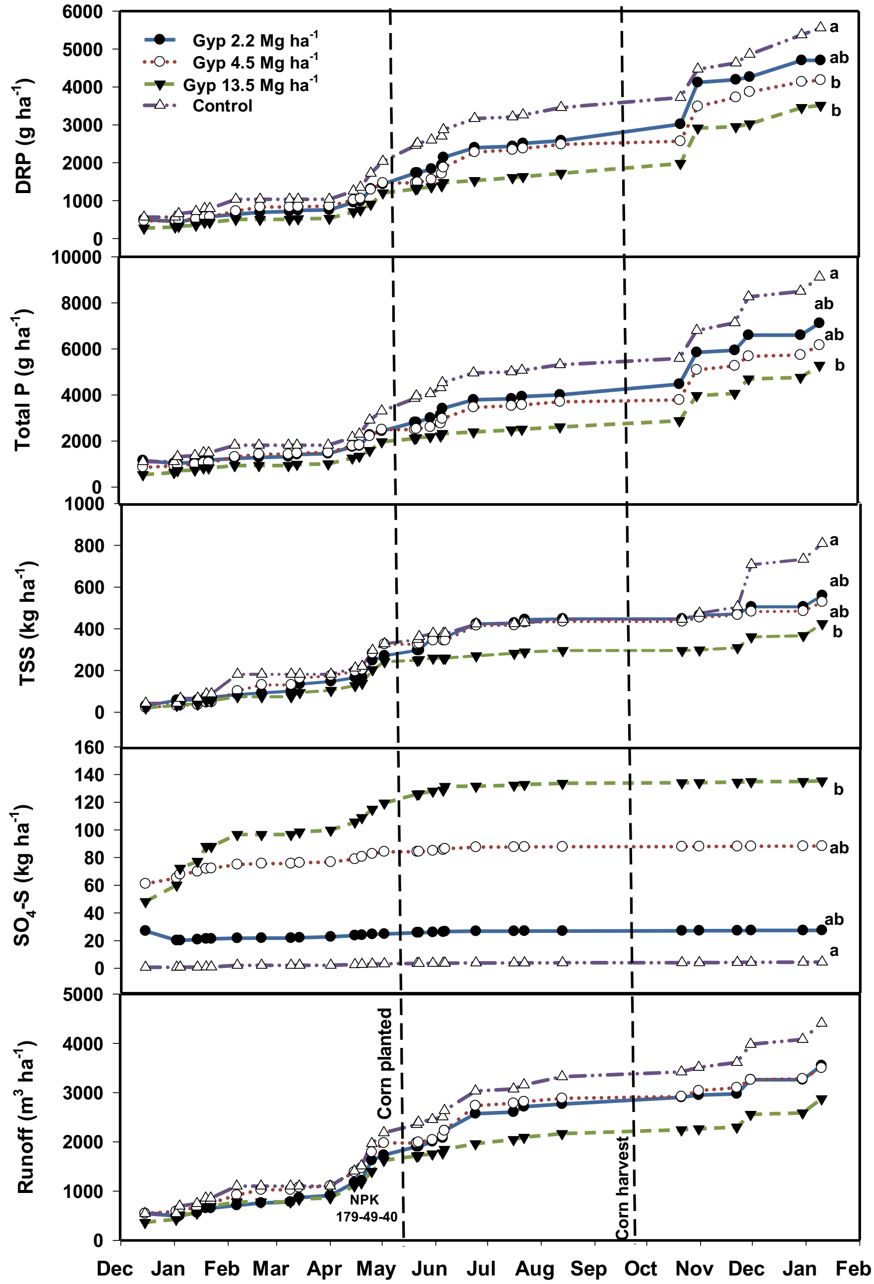

Fig. 2 Cumulative nutrient export and discharge from the runoff plots for 1-year crop rotation of corn-no crop with four treatments (FGD gypsum at rate 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, 13.5 Mg ha^{-1}, and control) (December 2018–January 2019). Treatments followed by a same letter are not statistically different ($\alpha = 0.1$)

in topsoil (Baker & Laflen 1983; Puustinen et al., 2005). Also, in reduced tillage conditions, the P content of the uppermost soil layer (0–15 cm) increases, and for deeper uncultivated layers it decreases. Moreover, the dead cells of crop residue on the soil surface have been found to release DRP (Johnson et al., 1977). High STP (> 67 kg ha^{-1}) level and P fertilization increased DRP losses in surface runoff and reduced effectiveness of gypsum in reducing soluble P in the soil.

The DRP:TP ratio in surface runoff in all the treatments ranged from 0.66 to 0.75, which showed P is primarily exported as DRP from the agriculture fields. Similarly, Buda et al. (2009) reported DRP:TP ratio averaging 0.70 in runoff at all landscape positions. Some studies have reported that distinct landscape positions affect the runoff generation process, which can influence DRP and TP concentration in runoff (Buda et al., 2009; Daryanto et al., 2017). Buda et al. (2009) found that seepage slopes and transitional midslope had greater DRP and TP concentration values than foot slope positions. Our study site was in the midslope landscape position (1.6% slope), which may have contributed to relatively high concentrations of DRP and TP in runoff. In addition, P sorption efficiency of FGD gypsum can vary with retention time and P concentration in water. In a laboratory flow-through experiment, FGD gypsum has been found to be poorly buffered (unless soil pH > 6) and require longer retention time for P removal compared to other Ca-rich P-sorbing materials (Stoner et al., 2012). The retention time following runoff initiation might not be sufficient in this study impacting P solubilization. However, average 6.5 soil pH showed P was removed via Ca precipitation from high P soil. Similarly, Torbert et al. (2005) observed initial effect of gypsum resulting in decreased P loss in runoff, but this effect was not long-lived. Overall, our results indicated that FGD gypsum application at the rate of 4.5 Mg ha^{-1} and 13.5 Mg ha^{-1} was most effective at reducing TP and DRP load in surface runoff in high STP soils. Further research is needed to evaluate the long-term binding capacity of FGD gypsum for reducing soluble P in surface runoff.

3.3 SO_4-S in Surface Runoff

Soil application of FGD gypsum resulted in increased SO_4-S concentration and load values ($P < 0.001$) in gypsum treatments than control during the initial post-gypsum application period and after fertilizer application time-period (Table 1). Gypsum application increased cumulative SO_4-S load significantly in 13.5 Mg ha^{-1} treatment by 30 times compared to control. In the events following treatment application, SO_4-S concentration values were relatively high, but in successive runoff events, they decreased with time. This increase in sulfate load was anticipated since the gypsum dissociates into calcium and sulfate following the application. The mean SO_4-S concentration and load values during the initial post-gypsum application period and post-fertilization periods were significantly higher in all gypsum treatments than in control. These mean concentration values were lower than USEPA-recommended limits (400 mg L^{-1}) (USEPA 1992).

3.4 Total Suspended Solids

Mean TSS concentration and loads ($P > 0.1$) were similar among gypsum treatments during the initial post-gypsum period and the post-fertilization period (Table 1). Cumulative TSS ($P = 0.1876$) load was reduced by 48% with 13.5 Mg ha^{-1} compared to control (Fig. 2). These are contrasting results compared to those by Truman et al. (2010) on Faceville loamy sand soils that showed 58% less sediment loss after gypsum application. In our study, there was considerable seasonal variation in TSS compared to DRP, which was relatively more stable seasonally. The probable underlying reasons behind the variation in TSS were changes in the hydrologic conditions and varying ground cover. During the crop season, all the treatments had lower sediment loss compared to control which is likely due to the presence of soil cover which reduced the impact of raindrop kinetic energy (Mannering & Meyer, 1963).

Reduced soil losses may also be due to elevated electrolyte concentration, which enhances clay flocculation and reduces sediment transport capacity of runoff by increasing soil infiltration (Miller 1987; Shainberg et al., 1989; Norton et al., 1993). Given there was no impact on sediment losses after gypsum application, this suggests gypsum may not reduce particulate P losses. Only a few studies have indicated that gypsum also decreases particulate P (Favaretto et al., 2006).

3.5 Heavy Metals in Soil and Runoff Water

The lab results of the applied FGD gypsum subsample showed the metals As, Cd, Cu, Pb, Hg, Mo, Ni, and Se were below the detectable limit (Martin et al., 1994). In the runoff event collected after treatment application (December 15, 2018), gypsum treatment 13.5 Mg ha^{-1} had a higher concentration of Cd, Mo, and Se in the runoff compared to control but was below the USEPA-recommended limit (Driscoll, 2002) (Table 2). Torbert and Watts (2014) showed that the concentration of heavy metals in runoff was below detection limits following gypsum application. For soil samples collected at depth of 0–15 cm, there was no significant increase in heavy metal content of

Table 2 Concentration of trace elements in the surface runoff following flue gas desulfurization (FGD) gypsum application, including the USEPA-recommended limits. Means within a column followed by the same letter are not statistically different ($\alpha = 0.1$)

Treatment	Concentration									
	As	Cu	Cd	Hg	Mo	Ni	Pb	Se	Zn	Cr
	μg L^{-1}									
USEPA	340	1300	1.8	1.4	70	470.00	82.00	50.00	5000.00	16.00
Control	1.7 abc	8.1	0.06 a	-	0.3 a	2.8 ab	4.3 ab	0.30 a	47.9	2.5
2.24 Mg ha^{-1}	1.8 b	6.2	0.07 a	-	0.5 ab	3.3 b	5.7 a	1.2 ab	76.2	3.2
4.48 Mg ha^{-1}	1.6 abc	5.2	0.08 a	-	0.5 a	2.7 a	3.5 b	2.4 b	93.2	2.4
13.45 Mg ha^{-1}	1.5 c	5.3	0.14 b	-	0.8 b	2.8 ab	3.3 b	4.5 c	185.9	4.5
p-value	0.07	0.5154	0.0924	-	0.1301	0.2062	0.1222	0.0023	0.341	0.3521

Note: *As* arsenic, *Cu* copper, *Cd* cadmium, *Hg* mercury, *Mo* molybdenum, *Ni* nickel, *Pb* lead, *Se* selenium, *Zn* zinc, *Cr* chromium

soil after treatment application (Table 3). The only metal that was above the EPA standard (18 kg ha^{-1}) was Ni, and soil Ni concentrations were all lower post-treatment compared to pre-treatment, which suggests that the gypsum did not add a significant amount of Ni to the soil; rather it was already present in the soil (USDA-NRCS, 2000) (Table 3). Following treatment application, arsenic was higher in all treatment and control plots, but it was well below the USEPA standard. In addition, Hg, Mo, and Cd were not detected in any of the treatment plots before and after gypsum application to soil. The year x treatment interaction was not significant ($P > 0.1$). Other studies reported mixed results about the soil contamination following FGD gypsum application. Chen et al. (2001) reported no soil contamination even at higher FGD gypsum application rates. In contrast, Chen et al. (2015) found a positive correlation of As and Hg concentrations in soils with FGD gypsum. At a depth of 0–20 cm, Ca, S, B, and Zn concentrations were increased, but there was no increase in any other trace metals when FGD gypsum was applied at a 280 Mg ha^{-1} application rate in an abandoned coal mine in Ohio (Chen et al., 2013). However, the concentration of these heavy metals in runoff water was found to be below USEPA water quality–recommended limits in all the treatment and control plots.

3.6 Plant Biomass and Soil Quality

The aboveground plant biomass samples indicated no change in uptake of Ca, Mg, and P, when the soil was treated with different rates of FGD gypsum (Table 4). The 4.5 Mg ha^{-1} gypsum treatment did

Table 3 Mean concentration values of trace elements in the soil (0–15 cm) pre-and post-flue gas desulfurization (FGD) gypsum application, including USEPA standards. Means within a column followed by the same letter are not statistically different ($\alpha = 0.1$)

Treatment	Year	As kg ha^{-1}	Cu	Cd	Hg	Mo	Ni	Pb	Se	Zn	Cr
USEPA		41	1500	39	300	17	18	420	100	2800	3000
Control		15.2	22.1	0.53	-	-	38.1	30.7	-	97.4	41.1
2.24 Mg ha^{-1}		15.2	24.7	0.53	-	-	51.8	31.8	-	101.1	40.5
4.48 Mg ha^{-1}		14.5	20.6	0.51	-	-	37.7	29.5	-	92.6	40.5
13.45 Mg ha^{-1}		15.2	21.6	0.52	-	-	39.7	30.7	-	96.6	40
p-value		0.8319	0.564	0.9666	-	-	0.3823	0.7413	-	0.2556	0.8026
	2018	12.1 a	23.8	0	-	-	47.6	35.2	-	100.8 a	41.3
	2019	17.9 b	20.7	1.05	-	-	36.0	26.2	-	93.1 b	39.7
p-value		<0.0001	0.1587	<0.0001	-	-	0.091	<0.0001	-	0.0161	0.0614

Note: *As* arsenic, *Cu* copper, *Cd* cadmium, *Hg* mercury, *Mo* molybdenum, *Ni* nickel, *Pb* lead, *Se* selenium, *Zn* zinc, *Cr* chromium

Table 4 Comparison of mean calcium (Ca), sulfur (S), magnesium (Mg) and phosphorus (P) and concentrations in plant biomass samples. Within a column, means followed by the same letter are not statistically different ($\alpha = 0.1$)

Treatment	Ca	S	P	Mg
	g kg^{-1}			
Control	2.9	0.6 b	3.7	1.1
2.2 Mg ha^{-1}	2.7	0.7 ab	3.6	1.7
4.5 Mg ha^{-1}	3.1	0.9 a	4.1	1.2
13.5 Mg ha^{-1}	2.1	0.7 ab	3.7	1.6
p-value	0.1239	0.054	0.8037	0.5063

have a significantly higher concentration of S in plant tissues compared to other treatments and control. Sulfur concentration in plant biomass was significantly higher in gypsum treatment 4.5 Mg ha^{-1}. In a study in Iowa, Sawyer et al. (2011) reported no effect of S fertilization on corn growth when S was applied at a rate or more than 0.04 Mg ha^{-1}. Similarly, in Ohio S application at 0.03 Mg ha^{-1} as FGD gypsum increased corn yield and positive NxS interaction was observed (Chen et al., 2008). As reported by the National Atmospheric Deposition Program (1999, 2019), in Illinois, annual deposition of S decreased from 18 kg ha^{-1} in 1997 to 8 kg ha^{-1} in 2016. Kim et al. (2013) indicated increased S uptake in corn for improved growth. However, if enough S is available via S mineralization in soil, then applied S will not be beneficial.

The mean concentrations of essential plant nutrients Ca (P = 0.1304), Mg (P= 0.2463), and P (P= 0.4304) in the soil in the 5–15-cm layer, were not affected after 1 year of treatment (Table 5). Applied Ca can move downward quickly from the topsoil. These results were consistent with other studies where Ca in gypsum leaches down quickly from the soil surface, but in the subsoil it can be strongly absorbed and retained for longer periods (Toma et al., 1999). Also, a significant year x treatment (P = 0.0688) interaction showed increased Ca content in 13.5 Mg ha^{-1} compared to control 5–15-cm soil depth. Mean S values were significantly different at 0–5 cm (P = 0.0002) and 5–15 cm (P =0.0111) soil depth when gypsum was applied at a rate of 13.5 Mg ha^{-1}. Kost et al. (2014) reported similar results in a gypsum study in Ohio where Mehlich-3 concentration of S was greater at 0–15 cm for the application

Table 5 Comparison of mean soil calcium (Ca), sulfur (S), magnesium (Mg), and Bray I phosphorus (Bray I P) concentrations at 0–5 and 5–15 cm of soil depth. Within a column means followed by the same letter are not statistically different ($\alpha = 0.1$)

Treatment	Year	Ca		Mg		S		Bray 1 P	
		0–5 cm	5–15 cm	0–5 cm	5–15 cm	0–5 cm	5–15 cm	0–5 cm	5–15 cm
		kg ha^{-1}							
Control		1102	2455 ab	72 a	152	8 b	11 b	66	33
2.2 Mg ha^{-1}		1061	2501ab	66 b	148	11 b	17 b	73	42
4.5 Mg ha^{-1}		1057	2421 b	61 b	133	14 b	25 b	68	38
13.5 Mg ha^{-1}		1164	2686 a	62 b	143	33 a	60 a	77	46
p-value		0.3283	0.1304	0.002	0.2463	0.0002	0.0111	0.5282	0.4304
	2018	1137	2657 a	75 a	154 a	8 b	13 b	80 a	47 a
	2019	1056	2374 b	56 b	134 b	25 a	43 a	62 b	33 b
p-value		0.077	0.0028	<0.0001	0.0067	<0.0001	0.0059	0.0077	0.0184
Control	2018	1147	2752 a	76 a	163 a	8 bc	12 b	78	42
2.2 Mg ha^{-1}	2018	1140	2715 ab	76 a	164 a	10 bc	11 b	86	47
4.5 Mg ha^{-1}	2018	1091	2442 abc	73 ab	135 b	6 c	12 b	79	44
13.5 Mg ha^{-1}	2018	1170	2719 ab	75 a	155 ab	7 c	15 b	77	54
Control	2019	1058	2158 c	68 b	142 ab	7 c	9 b	54	25
2.2 Mg ha^{-1}	2019	983	2286 c	57 c	131 b	12 bc	23 b	61	36
4.5 Mg ha^{-1}	2019	1024	2399 bc	50 d	132 b	21 b	37 b	58	31
13.5 Mg ha^{-1}	2019	1158	2653 ab	49 d	131 b	60 a	104 a	77	38
p-value		0.7060	0.0688	0.0089	0.4365	<0.0001	0.0192	0.4065	0.9752

rate of 20 Mg ha^{-1}. The main reason for significantly higher S values in the top layer of soil is attributed to gypsum addition.

There was a significant reduction of Mg in the top 0–5 cm of soil in all the gypsum application rates. Gypsum is a soluble source of Ca and electrolytes and provides the Ca ion to the soil exchange complex, where it has a greater affinity for exchange sites over Mg and Na ions in most soils (Shainberg et al., 1989; Dontsova & Norton, 2002). Higher loading of Ca likely resulted in a competitive exchange of these cations and subsequent leaching of Mg from the soil (Alva et al., 1998; Kost et al., 2014). The same results were reported by Kukier et al. (2001) that surface application of FGD gypsum significantly reduced exchangeable Mg by up to 90% at a depth of 60 cm below the surface of Pelham soil in Georgia following 13 months of gypsum application at rates of 5, 10, and 20 Mg ha^{-1}. No significant effect of treatment application was found on soil Bray I P values. FGD gypsum may have reduced water-extractable P as there was a P load reduction in surface runoff at all gypsum treatment levels during the initial post-gypsum application period. However, soil P values were higher in the 0–5-cm layer than the 5–15-cm layer, an indication of P stratification in the no-till soil of the study site. Some studies reported similar results, in which authors presented no effect on soil Mehlich 3 P when gypsum was applied to high STP level soils, but there was 37 to 57% reduction in water-extractable soil P (Stout et al., 2003; Torbert et al., 2018). Similarly, Stout et al. (1998) found FGD gypsum to be effective in reducing water-extractable P with little effect on soil pH, soil test P, or plant available P. As the pH range of soil averaged around 6.5, there was likely no Al and Fe toxicity found in these soils. Treatment application did not affect Al and Fe concentration in the upper layer (0–15 cm) of soil.

After 1 year of application of different rates of gypsum, no significant changes in soil physical properties were observed including aggregate stability (P = 0.6885) and infiltration rate (P = 0.5674). Bulk density (P = 0.0698) was significantly lower in gypsum treatment 4.5 Mg ha^{-1} (1.32 g cm^{-3}) by approximately 4% compared to control (1.37 g cm^{-3}) and other gypsum treatments (2.2 Mg ha^{-1} = 1.37 g cm^{-3}, 13.5 Mg ha^{-1} = 1.38 g cm^{-3}). Even though gypsum treatments resulted in lower runoff rates, gypsum application rates did not significantly increase soil infiltration rate or reduce soil bulk density. In this study, the average infiltration rates were 0.84, 0.93, 0.97, and 0.63 cm h^{-1} for the control, 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, and 13.5 Mg ha^{-1} gypsum treatments, respectively. Mean aggregate stability in the control, 2.2 Mg ha^{-1}, 4.5 Mg ha^{-1}, and 13.5 Mg ha^{-1} gypsum treatments was 436, 481, 470, and 486 g kg^{-1}, respectively. These results were consistent with other studies where FGD gypsum application resulted in runoff reduction but no significant differences in soil infiltration rates (Truman et al., 2010). No observed improvement in soil aggregate stability indicates that applied Ca had not strengthened bonds between soil particles enough to increase pore space 1 year after application as suggested by Presley (2016). Similarly, Buckley and Wolkowski (2014) reported no beneficial impact on soil physical properties of FGD gypsum applied at rates of 0, 1.12, 2.24, and 4.48 Mg ha^{-1}, 12 weeks after treatment application.

Although the use of gypsum is considered a standard practice for reclamation of sodic soils, there is less consistency demonstrated concerning improvement in the physical properties of non-sodic soils. However, long term-effects of gypsum application are found to be more pronounced on clayey loam soils, and its impact on soil physical properties may be observed in future years (Toma et al., 1999).

4 Conclusion

This study demonstrated that the use of FGD gypsum as a soil amendment practice may reduce the detrimental losses of P from corn-soybean plots having high STP values. Significant reductions in runoff discharge, DRP and TP load were observed in all gypsum treatments compared with the control before fertilizer application. Following fertilizer application, the effectiveness of gypsum in reducing dissolved P was diminished immediately. In watersheds with water quality concerns, our study results support the USDS-NRCS-recommended application rates of 2.2 Mg ha^{-1} and 4.5 Mg ha^{-1}, as they resulted in 35% and 45% reductions in DRP loads in surface runoff, respectively. The highest application rate (13.5 Mg ha^{-1}) resulted in only an additional 6.5% reduction beyond the 4.5 Mg ha^{-1} rate, which would not justify the extra cost and additional sulfur loading to the soil. FGD gypsum applications did not increase the concentration of potentially toxic heavy metals such as Hg or As in soil or surface runoff (Koralegedara et al. (2019)). Results suggest that applying FGD gypsum could be an effective practice to reduce P losses in surface runoff. FGD gypsum can be coupled with other in-field and edge of field practices to meet nutrient reduction goals. Additionally, nutrient management strategies such as timing and rate of fertilizer application based on soil tests and gypsum application rates need to be addressed. Further work must be conducted to determine the longevity of FGD gypsum effects on water quality without the confounding impacts of P fertilization and to assess its impact at the watershed scale.

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Data Availability The authors declare that all data supporting the findings of this study are available within the article.

Declarations

Conflict of Interest The authors declare no competing interests.

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