

CORN RESPONSE TO SULFUR IN THE RED RIVER VALLEY

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ABSTRACT

Sulfur (S) deficiency symptoms are becoming common to crops grown in the Red River Valley of North Dakota and Minnesota. Corn (*Zea mays* L.) response to incremental sulfate-S (0, 11, 22, 33, and 44 kg S ha⁻¹) was studied (n=12) during the 2018-2020 growing seasons in a series of experiments. Corn yield and S uptake did not respond to S fertilizer ($P \geq 0.05$) additions, but yield varied across sites. Ten out of 12 sites showed an increase in grain yield over control but not significant. Corn, spring wheat (*Triticum aestivum* L.), and sugar beet (*Beta vulgaris* L.) responses to S forms were also studied. Only spring wheat showed a significant ($P \leq 0.05$) response to S forms. Growers should follow the current recommendation to apply 11 kg S ha⁻¹ to compensate for the grain S removal and avoid grain yield loss to S in susceptible areas of fields.

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GENERAL INTRODUCTION

During the past few decades (1980-2015), corn (*Zea mays* L.) grain yield has substantially increased at the rate of 121 kg ha⁻¹yr⁻¹ in North America due to the use of improved genetics and management practices (Assefa et al., 2017). Modern corn hybrids for a grain yield of 12.0 Mg ha⁻¹ contain 286 kg ha⁻¹ nitrogen (N), 114 kg ha⁻¹ phosphorus (P), 202 kg ha⁻¹ potassium (K), and 26 kg sulfur (S), with grain harvest index values of P (79%), S (57%), and N (58%) (Bender et al., 2013). Since 1990, the northern Great Plains of the USA have undergone a shift in crop rotation from traditional small grain dominant rotations to rotations consisting of, or including corn and soybean [*Glycine max* (L.)] based rotation (O'Brien et al., 2020).

Partially due to increased area under corn-soybean rotation, which requires greater S availability compared with small grain dominant rotations. Additional pressure on S availability is the greater yields experienced by farmers continually through the past two decades, the greatly reduced S from precipitation, and a much wetter growing season in the northern Great Plains of the USA that results in greater spring S leaching, S deficiencies are becoming common during the early growth stages of corn across the northern Great Plains (Scherer, 2001; Erikson et al., 2004; Girma et al., 2005; Franzen, 2015). There are four million hectares of soils deficient in the Canadian Prairie provinces in plant-available S, and substantially greater areas are potentially S-deficient (Grant & Hawkesford, 2015). Increasing applications of high analyses fertilizers such as monoammonium phosphate (MAP), di-ammonium phosphate (DAP) and ammonium polyphosphate (APP) that contain little or no S have reduced the incidental application of S as a nutrient and has increased the demand for S as a fertilizer nutrient (Matamwa et al., 23018). Sulfur deficiency in crops is most common in soils that are low in soil organic matter (SOM),

where S release by mineralization is limited, and on the coarse-textured soils (sandy loams, loamy sands and sands), where S leaching from the rooting zone is dominant (Grant et al., 2012).

In the northern Great Plains, the primary S fertilizer sources are ammonium sulfate (AS) $[(\text{NH}_4)_2\text{SO}_4]$ (24% S), gypsum (CaSO_4) (18% S), and bentonite clay blended, finely ground elemental sulfur (ES) ($\approx 90\%$ S). Ammonium sulfate and gypsum fertilizers have plant-available sulfates (SO_4^{2-}); however, SO_4^{2-} is highly soluble and mobile in soils and prone to leaching (Chien et al., 2011). For this reason, the S deficiency may also be found on coarse to medium textured soils with high SOM that have greater S mineralization potential but whose mineralization rate is slowed by constant spring rainfall and surface S leaches below the present below crop rooting zone.

Field experiments have been conducted to examine corn, sugarbeet and spring wheat response to S fertilizers. In the northern Great Plains, the most widely used S sources are AS ammonium thiosulfate (ATS) $[(\text{NH}_4)_2\text{S}_2\text{O}_3]$ and various ES forms. Recently, fertilizer manufacturers have developed and are marketing monoammonium phosphate that contain AS and ES, or ES alone. These fertilizers have the advantage of more even distribution of S due to the greater number of granules per unit area compared with AS application at low rates alone. Also, some fertilizer retailers enjoy the luxury of not requiring a separate bin storage space for AS. However, ES is not available for plants until it is oxidized to $\text{SO}_4\text{-S}$ by S oxidizing bacteria, Thiobacillus (chemolithotrophs), or, more commonly in the northern Greater Plains, other bacteria or fungi (heterotrophs) that oxidize S much more slowly than Thiobacillus. The oxidation rate of ES particles increases with decreasing particle size of ES (Boswell & Friesen, 1993). Recently, some fertilizer companies have introduced fertilizers containing micronized ES particles ($<100 \mu\text{m}$). It is believed that micronized-S (MS) would be oxidized soon after

fertilizer disintegration. The most MS products contain a blend of S in the form of AS and MS-ES, at various ratios. Sulfate-S of AS would supply initial S demand at early growth stages, while oxidation of MST-S (Micronized sulfur technology) would provide available S at later growth stages (Casteel et al., 2019).

In the Red River Valley of North Dakota and Minnesota, S deficiency has been found to reduce crop yield (Kaur et al., 2019). It is imperative to determine crops response to S and suitable source of S. Main objectives are to determine (i) changes in corn grain yield and uptake to incremental applications of fertilizer S and (ii) corn, wheat, and sugar beet response to S forms during the 2018-2020 growing seasons under on-farm conditions.

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LITERATURE REVIEW

Sulfur in plants

Sulfur (S) is an essential element required for normal plant growth (Alway, 1940). It is considered a secondary macronutrient, following the primary macronutrients nitrogen (N), phosphorus (P), and potassium (K), but is needed by plants at levels comparable to P (Kovar & Grant, 2011). Plants require S for synthesis of amino acids such as cystine, cysteine and methionine, (Havlin et al., 2005). Additionally, S is required in the formation of vitamins, enzymes, chlorophyll and plays a vital role in basic plant functions like photosynthesis and N fixation (Brady & Weil, 2008). Sulfur concentration in most crop plants ranges between 10 g kg⁻¹ and 15 g kg⁻¹, although concentrations more than 30 g kg⁻¹ have been reported for crops grown under saline conditions (Duke & Reisenauer, 1986). The majority of S required by a plant is absorbed from the soil solution by roots in the form of the divalent sulfate anion, SO₄²⁻ (Barber, 1995). Due to the important role of S in plant nutrition, S deficiency is recognized as a major limiting factor in crop production (Beaton, 1966; Tabatabai, 1984; Havlin et al., 2005). Visual symptoms of S deficiency vary with crop type and the severity of the deficiency (Duke & Reisenauer, 1986). Sulfur deficiency symptoms include reduced plant growth and chlorosis of the younger leaves, beginning with interveinal yellowing that gradually spreads over the entire leaf area. Since S is immobile in the plant, the deficiency symptoms tend to occur first in younger leaves.

Sulfur cycle

Generally, the soil S pool is divided into two major groups: organic and inorganic S. The transformation of soil organic sulfur into inorganic sulfate form i.e., S mineralization and reverse of this process that is the incorporation of inorganic sulfate form into soil organic pool is

commonly referred to as the S immobilization. Both processes play an important role in the cycling of S in the soil.

Around 95-98% of total soil S exists in the organic form (Rehm & Clapp, 2008) and the remaining 2-5% in the inorganic form. In contrast to the inorganic form, organic S is immobile in nature until it is oxidized to the mobile sulfate form (SO_4^{2-}) that is available for plant uptake (Scherer, 2001). Since about 95% of total soil S is in organic form that comes from manures, and crop residues (Ghani, McLaren, and Swift, 1991; Nguyen & Goh, 1992). The mineralization of organic S from soil organic matter and crop residues becomes an important source of S for plant uptake. In well-drained surface soils, the amounts of sulfate are often too small to provide adequate Sulfur for plant growth so that plants may be largely dependent upon the conversion of soil organic S to SO_4^{2-} for satisfactory S nutrition.

Sulfur mineralization and immobilization

Sulfur mineralization depends upon the forms of organic S present in the soil. Both main groups of organic S compound, i.e., ester sulfate, which has C-O-SO₃ linkages and carbon bonded S, which has direct C-S linkages, are mineralized to sulfate form (Houghton and Rose, 1976; Fitzgerald et al., 1984). In addition to this initial S content of the residue, residue C:S ratio, temperature, the moisture content in the soil, texture of the soil, and addition of lime are important indicators of S mineralization in soil. These all factors affect the S mineralization and its availability in the soil for plant uptake.

Organic form

Organic S in soil organic matter occurs in two primary forms: ester sulfate and carbon-bonded S. Ester sulfates are mineralized more easily than C-bonded S because they are not as likely to become bonded covalently to humic compounds as is C-bonded S (McGill & Cole,

1981). Also, they bond with the fulvic acid component of humic compounds, which are more active and labile in nature (Saggar et al., 1981). But C-bonded S is bonded with humic acid, which is passive and non-labile in nature (Bettany et al., 1980). Carbon-bonded S is mineralized only when a significant amount of S-rich substrate is available. Ester sulfate mineralization does not require microbial activity because its mineralization is controlled by the extracellular activity of sulfohydrolase enzymes (McGill & Cole, 1981). Sulfohydrolase enzymes include sulfatase, which can exist and remain active for a longer period in the soil (Dodgson et al., 1982). Also, plant roots can hydrolyze the ester sulfates very quickly. Fuller et al. (1986) observed that ester sulfate is mineralized more rapidly in the mineral horizon than in the organic horizon because these enzymes can easily retain on the mineral horizon.

Environmental factors

Mineralization, as with all parts of the S cycle, is greatly influenced by environmental factors such as soil, temperature, and moisture. Under favorable conditions such as optimum temperature (25-30°C) and moisture (70% water holding capacity), S mineralization is at peak rate. In winters during cold, wet regions, temperature conditions are not conducive to microbial activity, and substrates are also not available (Williams, 1967). At that time, there will be more immobilization. However, mineralization rates peak in autumn and early spring when substrate availability and soil moisture are high, and the temperature is conducive for microbial activity (Randlett et al., 1992). Swank et al. (1985) reported higher rates of immobilization in winter and late spring. Strickland et al. (1984) observed that temperature strongly affects the mineralization of S in the soil. At 5°C, mineralization was only one-fifth of its level at 20 or 30 °C. This response is due to different enzymes achieving optimal activity levels at different temperatures. Williams (1967) reported that S mineralization increased markedly with increasing temperatures

at 10, 20, and 30 °C over a 64-day incubation period. It was significantly lower at 10 °C than at higher temperatures.

Extreme moisture conditions also affect the process of mineralization. Sulfur mineralization may be inhibited near saturation by retarding the mineralization of organic matter. Likewise, mineralization can be slowed by very low soil moisture levels that are below those necessary for microbial activity (Williams, 1967). Wang et al. (2005) study the influence of soil moisture (40% and 70% water holding capacity) on S mineralization. They found that after 28 days of incubation S mineralization was four times higher at 70% than at 40% water holding capacity of the soil.

Crop species and initial S content

Sulfur mineralization varied widely among crop species. Most of the variability in S mineralization can be accounted for by the difference in initial S concentration of crop residues. Janzen & Kucey (1988) observed that rape showed much higher S mineralization than lentil and wheat because rape has relatively high tissue S concentration. They found that there is a highly significant linear relationship with an R^2 of 0.95 between cumulative S mineralization by day 56 and initial S concentration. Also, the use of canola varieties residue enhances the S mineralization because these varieties have high initial S content.

Crop residue C:S ratio also plays an important role in S mineralization. Sulfur is mineralized when the crop residue C:S ratio < 200 and is immobilized if C:S ratio > 400. Jashandeep et al. (2018) conducted an experiment using corn and spring wheat residue that resulted in S immobilization due to wide C:S ratio of corn and spring wheat residue. Tabatabai & Chae (1991) conducted an experiment to compare the mineralization of S in soils amended with various types of sewage sludge, animal manures and plant materials (alfalfa, cornstalk, soybean

residue, and sawdust). They found that S mineralization rate is more in the case of sewage sludge and alfalfa as compared to animal manures and other plant materials. This is because of the relatively high C:S ratio of the animal manures and some of the plant material studied. Also, crop residues can tie up available S and could result in S deficiency in crops.

Application of lime

Calcium carbonates also contain S at higher levels than many noncalcareous materials, so S is released when there is weathering of soil that is rich in calcium carbonate. Several studies reported that inorganic sulfate form is released after the application of lime. Mineralization was increased when calcium carbonate was applied to 17 Australian soils in the laboratory (Williams, 1967). Ensminger (1954) and Neptune et al. (1975) also observed that sulfate was released in the soil after the application of lime. Application of lime enhanced mineralization by creating favorable conditions for the microbial activity in the soil. Ellett & hill (1929) found that the losses of sulfate from lysimeters were greater from limed soils than from unlimed soils. White (1959) performed an incubation study and observed that the addition of calcium carbonate increased the amount of soluble sulfate in some soils. Williams and Steinbergs (1964) noted that there was an increase in the uptake of S by oats grown in pot culture with the application of calcium carbonate.

Type of soil

Tabatabai & Chae (1991) found that the S mineralization during a 20-week incubation period depends on the soil used. Less mineralization occurs in soil higher in clay content as clay can protect some of the more easily decomposable organic compounds from rapid microbial breakdown through encrustation and entrapment (Anderson, 1979; Paul & Vanveen, 1978; Tisdall & Oades, 1982). Jashandeep et al. (2018) observed that the cumulative mineralized S was

more in Glyndon soils as compared to the Fargo soils because Fargo soils are more clayey in nature as compared to the Glyndon soils. Also, soil surface properties influenced the soil organic S pool. Kaolinitic clays may have lower ester sulfate levels as compared to the soils having high content of organic matter, because the exposed sites on kaolinites provide fewer sites for formation of ester sulfate linkages (Watwood et al., 1986).

Crops response to S fertilizer and its forms

Corn is an important crop grown in the US and plays an integral role in its economy growth. The United States ranks first in corn production in the world. Corn acreage has increased significantly during past few decades from 24 million hectares in 1983 to more than 36 million hectares in 2018 (USDA, 2018). Similarly, average corn yield increased from 3.9 Mg ha⁻¹ in 1980 to 10.0 Mg ha⁻¹ in 2018 (USDA, 2018) because of high yielding varieties and efficient management practices.

Sulfur has received more attention due to increasing areas of S deficiency since the enactment of the US-Clean Air Act in 1970 and its subsequent implementation to remove S from coal and oil-based industries. Soil S levels have decreased steadily as S removal, crop yields have increased, and deposition of SO₄²⁻-S via rainfall, fertilizer, and pesticides has decreased (Dick et al., 2008).

Many studies have been conducted to study the crop response to S fertilizers. Sawyer et al. (2011) reported a significant increase in corn yield at 17 of 20 fields in 2017 and 11 of 25 fields in 2008 in Iowa. Whereas Fawcett et al. (2016) found only two sites showed a significant increase in corn yield in Iowa. Corn yield increased with 11 kg S ha⁻¹ at two sites of Red River Valley of North Dakota and Minnesota in the 2016-2017 growing seasons (Kaur et al., 2019). Kaiser et al. (2019) found an increase in yield of hard red spring wheat on sandy soils with the

application of 8 kg S ha⁻¹. Kruger et al. (2014) reported an increase in soybean yield of about 134 kg ha⁻¹ across S fertilizer rates of 0 to 56 kg S ha⁻¹ in Minnesota in 2011 and 2012.

In northern Great Plains, S is normally applied either as elemental sulfur or as a SO₄²⁻ source. Plants can use elemental sulfur if it is converted to SO₄²⁻ form (Janzen & Bettany 1984a, 1984b). Hence, crop yield is maximized when soluble sulfate forms of S like ammonium sulfate, ammonium thiosulfate are used rather than elemental sources (Grant et al., 2004 and Solberg et al., 2006). In the northern Great Plains, sulfate and thiosulfate are generally recommended over elemental S because slow oxidation of elemental sulfur is not suitable to supply SO₄²⁻ form. So, for the selection of fertilizers, growers depend on the local dealers or consultants. Fertilizer industries come up with attractive demands and introduce high priced S products like micronized S, and combination of elemental S and SO₄²⁻S.

Most studies done by researchers which compare the SO₄²⁻S sources with elemental S, conclude that in the initial year of SO₄²⁻S application is more effective in increasing crop yield (Solberg et al., 2006). However, some studies show that residual S from elemental sulfur fertilizers increase the yield in subsequent crops because S become available over time (Solberg et al., 2006). Fox et al. (1964) showed that elemental sulfur was more effective in providing S during second year in perennial crops like alfalfa. Mc Caskill & Blair (1989) showed that 99% of S from the single superphosphate was released after 72 days, where superphosphate with elemental sulfur and only elemental sulfur took a year to release 54 and 23% of S, respectively. Thiosulfate also must be oxidized to SO₄²⁻S before being available to plants. Also, thiosulfate act as a nitrification inhibitor (Goos, 1985). Corn fertilized with urea ammonium nitrate and ammonium thiosulfate yielded significantly more grain than urea ammonium nitrate with single superphosphate (Graziano & Parenta, 1996). The author hypothesized that this may be an effect

of thiosulfate which inhibit nitrification and urea hydrolysis, and therefore reducing the risk of N leaching.

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CORN RESPONSE TO INCREMENTAL APPLICATIONS OF SULFATE-SULFUR

Abstract

Reports of sulfur (S) deficiency symptoms in corn (*Zea mays* L.) grown in the Red River Valley of North Dakota and Minnesota are increasing. Current soil tests methods do not predict the availability of S correctly. Field trials were conducted to determine corn yield and S uptake response to incremental applications of S (0, 11, 22, 33, and 44 kg S ha⁻¹) in the form of ammonium sulfate ((NH₄)₂SO₄). Corn yield and S uptake varied significantly between sites. Out of 12 sites, only two had the greatest numerical corn yield without S application. Corn grain S removal ranged between 11-17 kg S ha⁻¹ at harvest. Fertilizer-S (SO₄) application did not result in a significant yield response. The current recommendation of 11 kg S ha⁻¹ is probably appropriate given the necessary to reduce the risk of future S deficiency across this region not knowing how spring rains may contribute to S loss and compensate for the removal of S in grain due to the uncertainty of adequate plant available S.

Introduction

The USA is the largest producer of corn in the world (USDA, 2018). High corn production is important for the financial health of farmers and to meet the feed grain demand of world livestock production (Ort & Long, 2014). In North Dakota, the area under corn production has increased during the past few decades from 0.28 million ha in 1980 to more than 1.68 million ha in 2018 (USDA, 2018). Average corn yield in the USA has increased from 3.9 Mg ha⁻¹ in 1980 to 10.0 Mg ha⁻¹ in 2018 (USDA, 2018). This increase in yield is due to the continued release of high-yielding hybrids and improved management practices (Grassini et al., 2011).

Nutrient management plays an important role in maximizing profitable corn production (Amanullah & Fahad, 2018; Stewart & Roberts, 2012; Fageria et al., 2008). Sulfur (S) is often

considered the fourth essential nutrient for the optimum growth of plants. It is required for several plant functions including amino acid production, which are the building blocks of proteins, and chlorophyll (Franzen & Grant, 2008). Unlike nitrogen (N), phosphorus (P), and potassium (K), S has not been studied as extensively because S deficiency symptoms were not commonly present outside of very deep, low organic matter, sandy-textured soils (Rehm, 2005) until the past twenty years. A major reason for increasing S deficiencies is the enactment of policies associated with the Clean Air Act of 1970, which has resulted in a series of regulations, whose implementation has resulted in the reduction of S deposition from the air and rainfall (Dick et al., 2015). In the past, precipitation was an important source of S and added a significant amount of S to soils (Andraski & Bundy, 1990). But due to reduced S air emissions resulting from regulating air quality, S additions from rainfall are much less, which has resulted in increasing S deficiency (Franzen, 2015). Additional reasons for the increase in S deficiency include increased crop demand due to increased yield and the use of more concentrated phosphate fertilizers with fewer S contaminants (Scherer, 2001), and wetter spring conditions in the northern Great Plains of the USA since 1992.

Because of the increased frequency of S deficiencies, many recent studies have been conducted to investigate the response of corn to S fertilizers. In Iowa, Lang et al. (2018) reported an increase in corn yield of 2.4 Mg ha⁻¹ across five sites. Also, in Iowa, 17 of 20 targeted fields in 2017 and 11 of 25 random fields in 2008 showed a significant corn yield increase with S fertilization (Sawyer et al., 2011). In contrasting studies, Fawcett et al. (2016) found only two out of 12 sites showed corn grain yield response to S in Iowa. In the Red River Valley of North Dakota and Minnesota, Kaur et al. (2019) found corn yield increased with 11 kg S ha⁻¹ at two of ten sites in the 2016-2017 growing seasons. Kaiser et al. (2019) conducted two studies in

Minnesota and found that the application of 8 kg S ha⁻¹ increased hard red spring wheat (*Triticum aestivum* L.) yield on sandy soils. For soybean, yield increases of about 134 kg ha⁻¹ across S fertilizer rates of 0 to 56 kg S ha⁻¹ were reported across various soil series in Minnesota in 2011 and 2012 (Kruger et al., 2014). Application of gypsum (CaSO₄) at the rates of 16 kg S ha⁻¹ and 67 kg S ha⁻¹ increased the soybean yield by 4.8% and 11.6%, respectively, in Ohio. Application of gypsum at the rate of 16 kg S ha⁻¹ increased the alfalfa (*Medicago sativa* L.) yield by 5% in 2001 and 6% in 2002 (Chen et al., 2008).

In Nebraska, there was no response to applied S at all 11 sites with different soil textures. Wortmann et al. (2009) concluded that response to S was expected only in sandy textured soils, with SOM of more than 10 mg kg⁻¹, apparently able to be adequately fulfill plant S demand. One confounding factor in many studies, particularly in Nebraska, is that irrigation water has enough SO₄²⁻ that no supplemental S is necessary (Olson & Rehm, 1986).

Soil tests of available S and SOM are not useful to predict the probability of a positive S crop response (Franzen, 2015). Many soils in this region contain high concentration of native gypsum deeper in the soil profile. The presence of gypsum in the soils can result in inaccurate soil test results due to crystal grinding and faulty diagnosis of S deficiencies (Spencer & Freney, 1960; Franzen, 2018). Similarly, Fixen (1990) found that soil tests were not beneficial for determining S fertilization needs because of extrinsic additions of S to the fields, such as in P fertilizers. In the North Central Region of the USA, mono-calcium phosphate extraction is the most common method for sulfate-S analysis in the soil (Combs et al., 1998). However, the Ca⁺² in the extracting solution can ion-pair or complex with the extracted SO₄-S to produce a potential error in soil test S measurement. As an alternative soil analysis, paired plant tissue analysis from productive areas compared to relatively less productive areas within fields may be more useful to

diagnose S deficiency. Due to general lack of predictability of S deficiency or adequacy from soil testing, we cannot be sure about the current recommendation for S application or predict the need for S fertilization in crops prior to crop growth. The current North Dakota recommendation for S application is 11 to 22 kg S ha⁻¹ to support an optimal yield of corn (Franzen, 2018).

Field trials were conducted to evaluate the effect of S fertilization on corn in the Red River Valley of North Dakota and Minnesota during the 2018-2020 growing seasons. The objectives of this study were (i) to determine corn grain yield response to an incremental application of sulfate-S (SO₄-S) (0, 11, 22, 33, and 44 kg ha⁻¹) under different soil series in the Red River Valley (ii) to determine corn S uptake at V6 growth stage and at maturity.

Materials and methods

Corn yield response to S was examined at twelve sites in the Red River Valley of North Dakota and Minnesota during the 2018-2020 growing seasons (Fig. 1). Soil series and texture information of experimental sites are presented in Table 1. Initial soil nutrient concentrations and basic soil physical-chemical properties are presented in Table 2. Rainfall and mean air temperature data are given in Table 3 and Table 4, respectively. Weather data was collected from the closest weather stations to each site associated with the North Dakota Agricultural Weather Network for each growing season (NDAWN, <http://ndawn.ndsu.nodak.edu/>).

Field experiment

Five SO₄-S rates at the rates of 0, 11, 22, 33, and 44 kg S ha⁻¹ in the form of ammonium sulfate (NH₄)₂SO₄ were arranged in a randomized complete block design with four replicates. Fertilizers were broadcasted by hand and incorporated to a 10 cm depth using a field cultivator operated at 10 km hr⁻¹ before planting corn in May. The experimental plot length and width were 7.60 m and 3.35 m, respectively. The inter-row spacing was 0.56 m with six rows within the

experimental plot at all sites except at Walcott I and Walcott II, where it was 0.76 m with four rows within the experimental unit. DKC35-88RIB cultivar of corn was planted at a seeding rate of 85,000 plants ha⁻¹. Roundup Max™ [a.i. isopropylamine salt of glyphosate (N-phosphono methyl glycine)] at a rate of 25 ml L⁻¹ was applied at the V8 growth stage once to control weeds. In 2020, Laudis™ (a.i. tembotrione: 2-[2-chloro-4-(methylsulfonyl)-3-[(2,2,2-trifluoroethoxy) methyl] benzoyl]-1,3-cyclohexanedione) was applied at V6 growth stage once to control weeds. Fertilizer nitrogen (N), phosphorus (P), and potassium (K) were applied according to the North Dakota State University (NDSU) Extension recommendations (Franzen, 2018). Monoammonium phosphate (MAP, 11-52-0) was used to supply P at a rate of 70 kg ha⁻¹ at Ada, Sabin, Casselton,

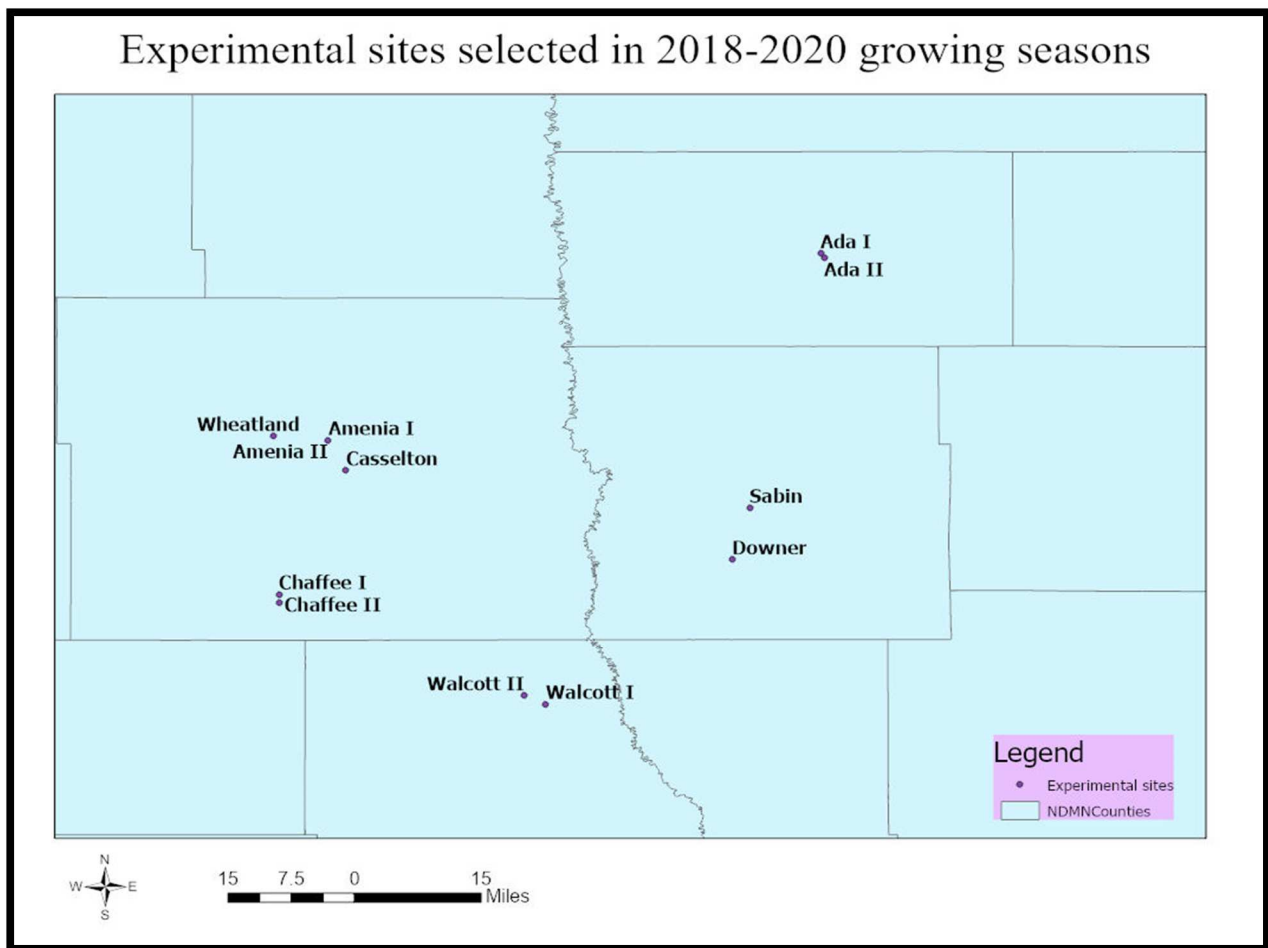


Figure 1. Location of experimental sites for the 2018-2020 growing seasons.

and Amenia and at a rate of 92 kg ha⁻¹ at the Walcott site in 2018. In 2019, MAP was applied at a rate of 45 kg ha⁻¹ at Ada, Amenia, Downer, and Chaffee and a rate of 92 kg ha⁻¹ at Walcott. During the 2020 growing season, MAP was applied at the rate of 70 kg ha⁻¹ at Chaffee and Wheatland. Muriate of potash (MOP, 0-0-60) was used to supply K at a rate of 80 kg ha⁻¹ at all the sites. Urea was used to supply N at a rate of 180 kg ha⁻¹. The N fertilizer application rate was adjusted so that a total rate of the 180 kg N ha⁻¹ was achieved, with consideration of residual soil N from entire experimental area, N supplied from the MAP rate, and the N contained in different S application rates. Corn was harvested in October in all years.

Soil analyses

Soil samples were collected before fertilizer application in May at a depth of 0-15 cm and 15-60 cm during the 2018-2020 growing seasons, using an auger. Soil samples were air-dried and ground to pass through a 2-mm sieve. Soil samples collected from 0-15 cm depth were analyzed for pH (1:1 soil/water) (Watson & Brown, 1998), electrical conductivity (Whitney, 1998), soil particle size distribution (Gee & Boudier, 1986), soil organic matter content by loss on ignition at 360°C (Combs & Nathan, 1998), plant available P index using the Olsen method (Frank et al., 1998), and plant available K index using 1-M ammonium acetate extraction (Warncke & Brown, 1998). Soil from each depth was extracted with 2 M potassium chloride (KCl) and analyzed for nitrate-N (NO₃-N) concentration (Gelderman & Beegle, 1998) and monocalcium phosphate for extractable sulfate-S (SO₄-S) (Combs et al., 1998). In-season soil samples were collected at the V6 crop growth stage at a depth of 0- 30 cm and were analyzed for available S. Ten grams of soil sample was extracted with 25 ml of monocalcium phosphate using charcoal to obtain clear filtrate and analyzed for S concentration using inductively coupled

plasma emission spectroscopy (ICP) (Thermo Scientific-ICAP 6500, Thermo Fisher Scientific, Waltham, MA, USA).

Plant sampling

Five random corn samples per experimental unit were collected from each experimental site at the V6 growth stage and again at corn harvest. The plants at V6 were cut off at the soil surface from rows not intended for grain harvest. The plants without grain and cob at maturity were also cut off at the soil surface and were located in rows not intended for grain harvest. Plant samples were dried at 60° C and then ground to pass through a 2-mm sieve. One-half gram of ground plant material was digested with 20 ml of concentrated nitric acid (Soltanpour & Havlin, 1980) and analyzed for S concentration using inductively coupled plasma emission spectroscopy. Sulfur uptake was calculated by the following equation.

$$\text{S uptake (kg ha}^{-1}\text{)} = \text{number of plants (ha}^{-1}\text{)} \times \text{average dry weight of five plants (kg)} \times \text{concentration of S (ppm)} / 10^6$$

(Equation 1)

Corn grain yield determination

The middle two rows from each experimental unit were hand-harvested to estimate the yield for all the sites during the 2018-2020 growing seasons. Grain moisture and test weight were measured using Dickey John Grain Moisture tester (GAC 500 XT, Illinois, USA). Grains were dried at 60° C and yields were adjusted after shelling and weighing to 155 g kg⁻¹ moisture. The yield was calculated using the following equation.

$$\text{Corn yield (kg ha}^{-1}\text{)} = \text{Weight of harvested corn (kg)} \times 10,000 \text{ m}^2 / \text{length of the row (m)} \times \text{width of the row (m)} \times (100\text{-grain moisture}) / 84.5$$

(Equation 2)

Statistical analysis

An overall ANOVA was performed using the PROC MIXED procedure in Statistical Analysis System 9.4 (SAS Inc., Cary, NC) for evaluating the site, year, treatment (S rate), and their interactions on corn yield and S uptake. Significant differences were determined at 0.05 significance level using least significant differences (LSD) within SAS.

Results

Location characteristics

Textural class and initial soil nutrient availability are presented in Tables 1 and 2, respectively. Plant available P index varied across all the sites; out of twelve sites, two sites, Walcott I and Walcott II, tested very low (0-3 mg kg⁻¹); five sites (Ada I, Amenia I, Sabin, Chaffee II, and Wheatland) were low (4-7 mg kg⁻¹); and the remaining five sites (Casselton, Ada II, Amenia II, Downer, and Chaffee I) tested medium (8-11 mg kg⁻¹). Plant available K index tested low for at Ada I and Downer (41-80 mg kg⁻¹), medium for four sites (Sabin, Ada II, Chaffee II, and Wheatland) (81-120 mg kg⁻¹), and very high for the remaining six sites (Walcott I, Amenia I, Casselton, Walcott II, Amenia II, and Chaffee I) (>151 mg kg⁻¹). Three sites tested relatively low in soil organic matter (SOM) (10-30 g kg⁻¹), four sites (Ada I, Sabin, Chaffee II, and Wheatland) tested medium in SOM (30-40 g kg⁻¹), and eight sites (Walcott I, Amenia I, Casselton, Ada II, Walcott II, Amenia II, Downer, and Chaffee I) tested very high (>40g kg⁻¹). Initial extractable nitrate-N (NO₃-N) and sulfate-S (SO₄-S) within 0-15 cm soil depth ranged from 11-40 kg ha⁻¹ and 7-22 mg kg⁻¹, respectively.

Total monthly precipitation and average air temperature for the twelve experimental sites during the 2018-2020 growing seasons are presented in Tables 3 and 4, respectively. The

Table 1. Soil series and texture information of twelve experimental sites selected in 2018-2020 growing seasons.

Site	Soil Series	Taxonomic classification¶
Ada I, MN	Augsburg sandy loam	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Typic Calciaquolls
Walcott I, MN	Bearden silt loam	Fine-silty, mixed, superactive, frigid Aeric Calciaquolls
Amenia I, ND	Glyndon sandy loam	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Sabin, MN	Lamoure sandy loam	Fine-silty, mixed, superactive, calcareous, frigid Cumulic Endoaquolls
Casselton, ND	Bearden clay loam	Fine-silty, mixed, superactive, frigid Aeric Calciaquolls
Ada II, MN	Augsburg sandy loam	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Typic Calciaquolls
Walcott II, ND	Bearden silt loam	Fine-silty, mixed, superactive, frigid Aeric Calciaquolls
Amenia II, ND	Glyndon sandy loam	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Downer, MN	Wyndmere loamy sand	Coarse-loamy, mixed, superactive, frigid Aeric Calciaquolls
Chaffee I, ND	Glyndon sandy loam	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Chaffee II, ND	Glyndon sandy loam	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Wheatland, ND	Bearden clay loam	Fine-silty, mixed, superactive, frigid Aeric Calciaquolls

¶Source: Web soil survey (<https://websoilsurvey.sc.egov.usda.gov/App/WebSoilSurvey.aspx>)

Table 2. Geographical locations and soil test information of twelve experimental sites selected in the 2018-2020 growing seasons.

	Latitude and Longitude	Planting date	Previous crop	Olsen-P mg kg ⁻¹	K	pH 1:1	EC dSm ⁻¹	OM g kg ⁻¹	NO ₃ -N† kg ha ⁻¹	SO ₄ -S mg kg ⁻¹
2018										
Ada I	47°19'41.9"N, 96°23'48.5"W	14 May	Spring wheat	5	67	8.5	0.48	24	20	18
Walcott I	46°30'52.4"N, 96°52'04.3"W	18 May	Soybean	3	188	8.0	1.76	39	22	13
Amenia I	46°59'05.7"N, 97°14'26.1"W	4 May	Soybean	5	385	8.0	0.96	48	24	10
Sabin	46°51'52.2"N, 96°31'5.80"W	2 May	Soybean	7	89	8.7	0.34	21	11	19
Casselton	46°56'52.2"N, 97°31'5.80"W	1 May	Soybean	11	253	7.4	0.48	49	39	7
2019										
Ada II	47°18'36.9"N, 96°23'26.5"W	8 May	Spring wheat	8	93	8.3	1.19	35	22	15
Walcott II	46°31'45.2"N, 96°54'14.3"W	12 May	Soybean	3	256	8.0	2.17	47	27	14
Amenia II	46°59'05.9"N, 97°14'26.4"W	10 May	Soybean	8	210	8.2	0.80	51	23	9
Downer	46°46'21.4" N 96°32'53.7"W	15 May	Sugar beet	11	70	7.5	0.68	35	25	16
Chaffee I	46°42'40.3" N 97°19'30.3"W	1 May	Sugar beet	11	193	7.9	0.53	42	28	11
2020										
Chaffee II	46°41'47.3" N 97°19'29.6"W	26 April	Sugar beet	6	96	8.6	0.30	12	22	22
Wheatland	46°59'30.2" N 97°20'00.9"W	31 May	Soybeans	4	118	8.0	1.23	32	40	8

† NO₃-N and SO₄-S from 0 to 60 cm, all other properties were determined from 0 to 15 cm.

Table 3. Total rainfall and departure from normal (1981-2010) (DN) for each site.

Site	May		June		July		August		September		October		Total rainfall	DN
	Total	DN	Total	DN	Total	DN	Total	DN	Total	DN	Total	DN		
mm														
2018														
Ada I	62.7	-19.6	78.2	-35.6	62.5	-30.7	66.6	-3.00	73.7	6.70	80.0	23.1	424	-59.1
Walcott I	22.1	-54.6	95.0	2.30	107	24.4	96.0	33.3	38.6	-24.9	46.2	-7.10	405	-26.6
Amenia I	53.9	-23.7	79.3	-21.0	65.3	-22.6	78.5	11.9	70.9	5.36	66.6	4.87	415	-45.2
Sabin	13.8	-66.4	148	43.1	117	35.5	91.9	24.1	63.0	-11.7	70.9	4.86	505	29.5
Casselton	53.9	-23.7	79.3	-21.0	65.3	-22.6	78.5	11.9	70.9	5.36	66.6	4.87	415	-45.2
2019														
Ada II	62.4	-19.9	68.3	-45.5	103	9.78	93.7	24.1	106	38.9	92.2	35.3	526	42.9
Walcott II	71.6	-5.10	67.3	-25.4	160	77.4	63.5	0.76	147	83.5	69.3	15.9	579	147
Amenia II	59.9	-17.5	121	20.7	156	68.1	102	35.4	147	81.5	76.9	15.2	663	203
Downer	62.4	-19.9	68.3	-45.5	103	9.78	93.7	24.1	106	38.9	92.2	35.3	526	42.7
Chaffee I	59.9	-17.5	121	20.7	156	68.1	102	35.4	147	81.5	76.9	15.2	663	203
2020														
Chaffee II	22.1	-54.6	75.7	-17.0	161	78.3	116	52.9	16.5	-47.0	9.90	-43.4	401	-30.8
Wheatland	41.1	-36.3	79.0	-21.3	122	34.9	116	49.1	13.2	-52.3	7.60	-54.1	379	-80.0

Table 4. Average air temperature and departure from normal (1981-2010) (DN) for each sites.

Site	May		June		July		August		September		October	
	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN
°C												
2018												
Ada I	16.7	3.47	20.6	2.06	20.6	-0.40	19.4	-0.85	13.3	-1.24	3.90	-2.91
Walcott I	17.2	3.25	20.6	1.54	20.6	-1.03	19.4	-1.15	13.9	-1.32	3.90	-3.80
Amenia I	16.7	3.26	20.6	1.92	20	-1.30	19.4	-0.98	13.9	-0.92	3.90	-3.39
Sabin	17.8	3.49	21.1	1.82	21.1	-0.9	19.4	-1.79	14.4	-1.07	4.40	-3.07
Casselton	16.7	3.26	20.6	1.92	20	-1.30	19.4	-0.98	13.9	-0.92	3.90	-3.39
2019												
Ada II	11.1	-2.12	18.3	-0.24	21.1	0.09	18.3	-1.95	15.0	0.46	5.00	-1.81
Walcott II	11.1	-2.84	19.4	0.34	21.7	0.07	18.9	-1.65	16.1	0.87	5.00	-2.07
Amenia II	10.6	-2.83	18.9	0.22	21.7	0.39	18.3	-2.08	15.6	0.78	4.40	-2.89
Downer	11.1	-2.12	18.3	-0.24	21.1	0.09	18.3	-1.95	15.0	0.46	5.00	-1.81
Chaffee I	10.6	-2.83	18.9	0.22	21.7	0.39	18.3	-2.08	15.6	0.78	4.40	-2.89
2020												
Chaffee II	11.9	-3.77	21.4	4.16	22.2	1.01	20.0	-1.05	13.9	-2.39	3.20	-8.09
Wheatland	12.0	-2.61	21.3	4.79	22.1	1.46	20.4	-0.03	14.1	-1.37	3.40	-7.01

cumulative rainfall from May through October was greater in 2019 (2957 mm) as compared with 2018 (2164 mm) and 2020 (780 mm) at all sites. In 2018, all sites received less cumulative rainfall than the 30-yr normal (1981-2010) except for Sabin. Sabin received 29.5 mm more cumulative rainfall from May through October than normal, whereas Ada I, Walcott I, Amenia I, and Casselton received 59.1, 26.6, 45.2, and 45.2 mm less than the normal, respectively. In 2018, all sites were drier than normal in May, and most of the precipitation occurred in August except for Ada I and Sabin. In 2019, the cumulative rainfall from May through October for all sites was much greater than the 30 yr. normal rainfall. The actual annual rainfall was 42.9, 147, 203, 42.7, and 203 mm more than normal for Ada II, Walcott II, Amenia II, Downer, and Chaffee I, respectively. In 2019, a dry period occurred in May at all the sites, while most of the rainfall occurred in September. In 2020, the cumulative rainfall from May through October for all the sites was also much less than the 30 yr. normal rainfall. The actual annual rainfall was 30.8 and 80.0 mm less than the normal rainfall for the sites Chaffee II and Wheatland, respectively.

Corn grain yield

Growing season, site, and their interaction affected corn grain yield; however, grain yield was not affected by S application rates and its interactions with year and site (Table 5). Across three growing years, corn grain yield was significantly greater in 2019 (15.4 Mg ha⁻¹) than in 2018 (14.5 Mg ha⁻¹) and 2020 (12.8 Mg ha⁻¹) (Table 6). Across 12 site-year, Walcott II in 2019 had the greatest site average yield (19.8 Mg ha⁻¹), and the lowest yield of 11.3 Mg ha⁻¹ was recorded at Amenia I in 2018. Sulfur application had no significant effect on grain yield across 12 site-year.

Table 5. Significance for grain yield, S uptake at V6 stage, stover S uptake at maturity, grain S uptake and total (stover + grain) S uptake at maturity at each site during the 2018-2020 growing seasons.

Source of variation	Grain yield	S uptake at V6 stage	Stover S uptake at maturity	Grain S uptake	Grain +Stalk
Site	<0.0001*	<0.0001*	<0.0001*	<0.0001*	<0.0001*
Year	<0.0001*	<0.0001*	0.03*	NS	NS
Treatment	NS	NS	NS	NS	NS
Site x year	<0.0001*	<0.0001*	<0.0001*	<0.0001*	NS
Year x treatment	NS	NS	NS	NS	NS
Site x treatment	NS	0.02*	NS	NS	NS
Site x year x treatment	NS	NS	NS	NS	NS

*Significant at 0.05 probability level; NS = non-significant

Table 6. Corn grain yield in response to an incremental application of sulfur at each site during the 2018-2020 growing seasons.

Treatment	2018					2019					2020				
	Ada I	Walcott I	Amenia I	Sabin	Casselton	Mean	Ada II	Walcott II	Amenia II	Downer	Chaffee I	Mean	Chaffee II	Wheatland	Mean
SO ₄ -S kg ha ⁻¹															
0	17.3 (1.28)	16.0 (0.68)	12.2 (0.98)	14.5 (0.96)	14.9 (1.72)	15.0 (1.12)	14.0 (0.54)	19.2 (1.80)	13.9 (1.85)	14.2 (0.53)	12.3 (1.69)	14.7 (1.28)	13.8 (1.20)	12.4 (0.73)	13.1 (0.97)
11	16.3 (0.71)	17.0 (0.67)	12.4 (1.31)	14.4 (0.40)	13.1 (0.76)	14.6 (0.77)	14.9 (1.60)	19.1 (1.51)	14.9 (1.19)	14.8 (1.24)	12.7 (1.15)	15.3 (1.34)	14.0 (3.71)	10.4 (2.61)	12.2 (3.16)
22	16.2 (0.72)	17.0 (0.71)	10.9 (1.08)	12.6 (2.13)	14.3 (1.37)	14.2 (1.20)	13.6 (0.53)	20.4 (0.49)	14.7 (1.15)	15.2 (1.02)	13.7 (0.31)	15.5 (0.70)	15.0 (1.65)	12.5 (1.16)	13.7 (1.41)
33	17.7 (0.77)	16.2 (0.43)	10.3 (0.61)	13.3 (1.95)	12.1 (1.73)	13.9 (1.10)	14.6 (0.76)	21.6 (1.30)	15.5 (0.82)	14.1 (0.38)	13.6 (0.93)	15.9 (0.84)	13.5 (1.76)	11.3 (2.72)	12.4 (2.24)
44	17.0 (0.75)	17.1 (0.36)	10.7 (1.51)	13.9 (1.08)	13.3 (1.21)	14.4 (0.98)	14.3 (1.01)	18.7 (1.08)	16.0 (1.70)	14.4 (0.81)	12.9 (1.60)	15.3 (1.24)	13.3 (1.70)	11.1 (1.96)	12.2 (1.83)
Mean	16.9 b [†] (0.85)	16.7 b (0.57)	11.3 g (1.10)	13.7 ef (1.30)	13.5 ef (1.36)	14.4 (1.04)	14.3 ecd (0.89)	19.8 a (1.23)	15.0 c (1.34)	14.6 cd (0.80)	13.1 f (1.14)	15.3 (1.08)	13.9 ed (2.00)	11.5 g (1.84)	12.7 (1.92)
Annual	14.4 (1.04) B [‡]					15.3 (1.08) A					12.7 (1.92) C				

[†] Means with different lowercase letters are significantly different at $P \leq 0.05$ by the LSD test.

[‡] Means with different uppercase letters are significantly different between two growing seasons at $P \leq 0.05$ by the LSD test.

Sulfur uptake at V6 stage

Sulfur uptake at V6 stage was significantly influenced by site, year, site \times year, and site \times S-treatment; however, S treatment had no significant effect (Table 5). Over two years, the 2019 growing season had higher S uptake (1.73 kg ha^{-1}) at V6 than in 2020 (1.00 kg ha^{-1}). Based on site-average, Chaffee I in 2019 had the highest V6-S uptake (3.26 kg ha^{-1}), significantly higher than the rest of the sites, and the lowest was observed at Ada II in 2019 (0.71 kg ha^{-1}) (Table 7).

Comparing S uptake in 2019 and 2020, the S uptake in 2019 had greater S uptake at V6 than 2020. Based on site-year, Chaffee I in 2019 had the greatest uptake, compared to the other sites. In 2019, average S uptake had the following sequence: Chaffee I > Walcott II > Amenia II > Downer > Ada II. In 2020, Chaffee II and Wheatland did not differ in S uptake at V6.

Stover S uptake at maturity

The site, year, and their interaction had a significant effect on stover-S uptake at maturity (Table 5). S-treatment had no effect on S uptake. Average stover-S uptake was higher in 2020 than in 2019 (Table 8). Across seven site-years, Wheatland, in 2020, had the highest stover-S uptake (average of all S-treatments) significantly greater than average stover S uptake of Chaffee II site. Within 2019, Walcott II, Amenia II, Ada II, and Downer had similar stover S uptake, and Chaffee I had the lowest stover S uptake, significantly lower than the rest of the sites. Stover S uptake ranged between $4.05\text{-}8.96 \text{ kg S ha}^{-1}$. For all seven sites, the highest S uptake was recorded in a treatment receiving S, not the control, but the increase in stover S uptake was inconsistent over S rates and was not significant at the 95% level.

Grain S uptake

Grain S uptake was only influenced by the site and its interaction with a year (Table 5). In 2019, Walcott had the highest grain S uptake, significantly higher than the rest of the four

Table 7. Sulfur uptake at V6 stage of corn growth in response to an incremental application of sulfur at seven sites during the 2019-2020 growing seasons.

Treatment	2019						2020		
	Ada II	Walcott II	Amenia II	Downer	Chaffee I	Mean	Chaffee II	Wheatland	Mean
SO ₄ -S kg ha ⁻¹	kg ha ⁻¹								
0	0.67 (0.07) a†	2.05 (0.34) bc	1.28 (0.21) defg	0.99 (0.15) adef	3.65 (1.00) h	1.73 (0.35)	0.77 (0.30) ad	0.99 (0.23) adef	0.88 (0.27)
11	0.69 (0.08) a	2.16 (0.27) b	1.44 (0.42) efg	0.86 (0.14) ad	3.71 (0.82) h	1.77 (0.35)	0.98 (0.40) adef	1.03 (0.26) adef	1.01 (0.33)
22	0.75 (0.15) a	2.27 (0.52) b	1.40 (0.17) efg	1.01 (0.26) adef	3.42(1.09) hi	1.77 (0.44)	1.17(0.21) adefg	1.20 (0.26) adefg	1.19 (0.24)
33	0.69 (0.09) a	2.28 (0.48) b	1.49 (0.21) fg	1.11(0.16) adef	3.12 (0.56) i	1.74 (0.30)	0.94 (0.22) ade	0.81 (0.30) ad	0.88 (0.26)
44	0.73 (0.08) a	2.46 (0.35) b	1.64 (0.10) cg	1.03 (0.09) adef	2.42 (0.08) b	1.66 (0.14)	1.38 (0.50) efg	0.75 (0.23) a	1.07 (0.37)
Mean	0.71 (0.09) e	2.24 (0.39) b	1.45 (0.22) c	1.00 (0.16) d	3.26 (0.71) a	1.73 (0.32)	1.05 (0.33) d	0.96 (0.26) de	1.00 (0.29)
Annual	1.73 (0.32) A‡						1.00 B		

† Means with different lowercase letters are significantly different for the same site at $P \leq 0.05$ by the LSD test.

‡ Means with different uppercase letters are significantly different between two growing seasons at $P \leq 0.05$ by the LSD test.

Table 8. Stover sulfur uptake at maturity of corn in response to an incremental application of sulfur at seven sites during the 2019-2020 growing seasons.

Treatment	2019						2020		
	Ada II	Walcott II	Amenia II	Downer	Chaffee I	Mean	Chaffee II	Wheatland	Mean
SO ₄ -S kg ha ⁻¹	kg ha ⁻¹								
0	5.77 (1.60)	5.68 (0.72)	6.07 (0.95)	6.06 (0.96)	4.05 (0.75)	5.53 (1.00)	4.24 (0.65)	8.14 (0.42)	6.19 (0.54)
11	5.54 (1.93)	6.74 (0.91)	5.29 (1.03)	5.42 (1.08)	4.10 (0.94)	5.42 (1.18)	4.98 (0.55)	8.96 (2.27)	6.97 (1.41)
22	6.25 (1.76)	6.40 (0.82)	5.79 (1.08)	6.31 (1.27)	3.99 (1.16)	5.80 (1.22)	4.76 (1.31)	6.60 (1.53)	5.68 (1.42)
33	5.69 (1.71)	5.95 (0.74)	6.20 (0.77)	6.84 (2.17)	4.00 (1.36)	5.74 (1.35)	5.51 (0.66)	8.83 (1.06)	7.17 (0.86)
44	6.34 (2.22)	5.96 (0.47)	7.37 (1.21)	5.84 (0.80)	4.42 (0.34)	5.99 (1.01)	6.02 (0.90)	8.19 (1.34)	7.11 (1.12)
Mean	5.97 (1.84) b†	6.15 (0.73) b	6.14 (1.01) b	6.09 (1.26) b	4.11(0.91) d	5.69 (1.15)	5.10 (0.81) c	8.14 (1.32) a	6.62 (1.07)
Annual	5.69 (1.15) B‡						6.62 (1.07) A		

† Means with different lowercase letters are significantly different for the same site at $P \leq 0.05$ by the LSD test.

‡ Means with different uppercase letters are significantly different between two growing seasons at $P \leq 0.05$ by the LSD test.

Table 9. Grain sulfur uptake in response to an incremental application of sulfur at seven sites during the 2019-2020 growing seasons.

Treatment	2019						2020		
	Ada II	Walcott II	Amenia II	Downer	Chaffee I	Mean	Chaffee II	Wheatland	Mean
SO ₄ -S kg ha ⁻¹	kg ha ⁻¹								
0	12.0 (0.63)	17.0 (2.91)	12.0 (1.38)	11.0 (0.08)	11.3 (1.84)	12.7 (1.37)	15.7 (5.60)	15.5 (2.28)	15.6 (3.94)
11	12.5 (1.91)	16.9 (0.34)	13.3 (0.75)	11.7 (1.50)	11.4 (1.22)	13.2 (1.14)	14.0 (3.56)	12.9 (3.61)	13.4 (3.59)
22	12.1 (0.52)	17.7 (1.33)	13.2 (1.76)	12.2 (0.99)	11.9 (0.61)	13.4 (1.04)	15.5 (3.71)	15.6 (2.00)	15.6 (2.86)
33	12.3 (1.14)	17.2 (1.63)	13.6 (0.88)	11.1 (0.33)	12.0 (1.61)	13.2 (1.12)	13.1 (0.76)	13.5 (3.26)	13.3 (2.01)
44	12.1 (0.66)	17.0 (0.42)	14.4 (1.62)	11.9 (0.90)	11.5 (1.52)	13.4 (1.02)	12.9 (2.43)	14.2 (2.91)	13.6 (2.67)
Mean	12.2 (0.97) cd†	17.1 (1.33) a	13.3 (1.28) bc	11.6 (0.76) d	11.6 (1.36) d	13.2 (1.14)	14.3 (3.21) b	14.3 (2.81) b	14.3 (3.01)
Annual	13.2 (1.14)						14.3 (3.01)		

† Means with different lowercase letters are significantly different for the same site at $P \leq 0.05$ by the LSD test.

sites (Table 9). Downer had the lowest grain S uptake, similar to Ada and Chaffee. Grain S uptake ranged between 11.1 to 17.7 kg S ha⁻¹.

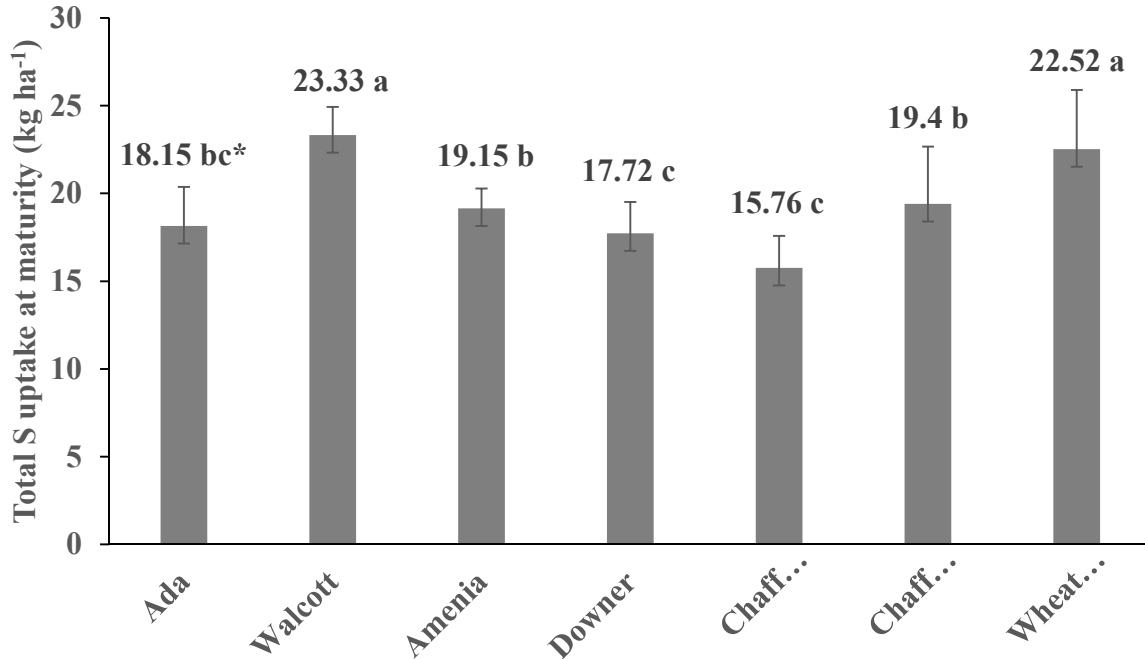


Figure 2. Mean total (stover+grain) sulfur (S) uptake at physiological maturity averaged across sites during 2019 and 2020 growing seasons.

* Different small case letters indicate significant differences at 95% confidence level.

Total (stover + grain) S uptake at maturity

Total aboveground S uptake varied significantly with the site. In 2019, Walcott had the highest total S uptake, significantly greater than the rest of the four sites; the lowest S uptake was observed at Downer, similar to Ada and Amenia (Fig. 2). In 2020, Wheatland had significantly highest S uptake than Chaffee. Total S uptake ranged between 15.8 to 23.3 kg S ha⁻¹. An increase in total S uptake to S addition over control was observed at all sites except Wheatland in 2020.

Discussion

The outcomes from on-farm trials from 2018-2020 to determine corn response to S indicate that corn yield and S uptake were more responsive to growing season and site characteristics rather than fertilizer- S application rate. Previous research (Kaur et al., 2019) also

found a lack of response to S and suggested that S from soil organic matter (SOM) and mineralization of residues in some soils was enough for crop growth and hence, applied $\text{SO}_4\text{-S}$ fertilizer had no effect on corn yield. Steinke et al. (2015) found that fine-textured soils in Michigan, USA, with relatively high SOM ($> 28 \text{ g kg}^{-1}$) and residual S $> 6\text{-}8 \text{ mg kg}^{-1}$ was enough to obtain maximum corn yield without application of S. Kim et al., (2013) found that yield response was not related to soil test $\text{SO}_4\text{-S}$ and the probability and magnitude of the response decreased with increasing SOM concentration in their Minnesota experiments. They found that yield response was greatest when SOM concentration was $< 20 \text{ g kg}^{-1}$, less between $20\text{-}40 \text{ g kg}^{-1}$, and was not responsive when $> 40 \text{ g kg}^{-1}$. Kaiser and Kim (2013) observed that grain yield response to S in Minnesota, USA, was recorded only when SOM concentration was $< 20 \text{ g kg}^{-1}$. Franzen & Grant (2008) wrote that S deficiency in the northern Great Plains is highly affected by soil properties. In soils low in SOM, less S is released through mineralization, resulting in a greater likelihood of S deficiency.

In this study, SOM concentration ranged between $12\text{-}51 \text{ g kg}^{-1}$, but grain yield at none of these 12 sites showed a significant response to $\text{SO}_4\text{-S}$ application. These findings suggest that neither SOM concentration nor $\text{SO}_4\text{-S}$ soil test could predict corn response to S. Along with the SOM, the texture of soil may also affect the S availability, as would presence or absence of early spring rainfall that would increase S leaching. Many previous studies have found that crops grown on sandy soil show more response to the application of S than those with greater clay content. Rehm (2005) conducted a study in central, south-central, and southeastern Minnesota and observed that the application of S fertilizer increased corn grain yield on sandy soils. The optimum rate of S fertilizer ranged from 6.7 to $13.4 \text{ kg S ha}^{-1}$, but the optimum rate of S fertilizer varied with site and year. They concluded that sandy soils with low SOM content ($< 20 \text{ g kg}^{-1}$)

are more responsive to S fertilizer due to inadequate release of S from SOM via mineralization. Also, sulfates leach more readily in sandier soils, contributing to generally less available S after spring thaw and during the growing season after high rainfall events. But soils of North central Region contain gypsum deeper in the soil profile. Fargo and Bearden soil series contain traces to several percent gypsum below 30 cm of soil depth (USDA-NRCS Web Soil Survey, <https://websoilsurvey.sc.egov.usda.gov/>), which can meet the plant S demand. During the summer season, due to high evapotranspiration demand, dissolved gypsum (SO₄-S) from the subsurface of soil moves upward with soil water, and S becomes available for uptake by plants (Nachshon et al., 2013). In this study, none of these 12 sites showed a significant response to SO₄-S application having sandy loam, clay loam, and loamy sand textures. However, under normal moisture conditions, corn roots can reach the zone of gypsum accumulation by the V6 to V8 growth stage and access plant available S below a 30 cm soil depth.

Crop residue S mineralization is an important process to fulfill plant S demands during the growing season (Kaur et al., 2018). Sulfur mineralization from previous crop residues may affect crop availability of S. This may be a major reason that corn does not respond to the application of S. More S mineralization was noticed for spring wheat residue with Fargo silty clay loam soils (Kaur et al., 2018). This may be due to the decomposition of crop residue at a rate greater than the mineralization of SOM. Hence, S mineralization is an important process that contributes S from mineralization during the crop cycle, but this is not generally considered during S analysis (Carciochi et al., 2019). This process should be considered as a way to adequately predict S availability for crops (O'Leary & Rehm, 1991) and it would improve the diagnosis of S deficiencies in crops (Carciochi et al., 2019).

Aula et al. (2019) found that S use efficiency of cereal crops around the world to be 18%. Low S use efficiency of cereals was mostly attributed to the leaching of SO₄-S from soil (Carciochi et al., 2019; Riley et al., 2002). In a sandy loam soil, (Riley et al., 2002) determined that 72% of fertilizers were leached, and none of the fertilizer S from the ammonium sulfate source remained in the experiment. Low adsorption of SO₄-S in soil with pH greater than 6 and lack of immobilization of SO₄-S to organic S are the two main reasons behind low S retention capacity in temperate climate soils with a positive cation exchange capacity and very low anion exchange capacity.

Kurbondski et al. (2019) observed that S increased plant mass, plant S concentration, and S uptake at the V8 stage and leaf S concentration at the R2 stage but did not increase corn grain yield. They found that their increase in S concentration was due to applied S fertilizer available for uptake. Similar results were observed in this study. Corn S uptake significantly increased at the V6 stage with the application of S but did not affect corn grain yield and total S uptake at maturity.

Conclusion

The S application did not increase corn yield. For most sites, S availability from mineralization might be enough to optimize yield. Yield and S uptake varied across site and year. As a standard method of available soil, S does not give a reliable estimate of S availability. Growers should apply the current recommendation of 11-22 kg S ha⁻¹ to reduce the chance of yield loss and compensate for the removal with grain. Further studies should focus on identifying site characteristics as well as deeper soil sampling (≥ 30 cm) to predict yield response besides SOM. Soil organic matter is not a reliable predictor for corn response to S in the Red River Valley of North Dakota and Minnesota.

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DO CROPS' RESPONSE TO SULFUR VARY WITH ITS FORMS?

Abstract

The most common forms of sulfur (S) fertilizers in the northern Great Plains are ammonium sulfate (AS), ammonium thiosulfate (ATS), and elemental sulfur (ES). Among these, AS is preferred over the others because of its readily available sulfate (SO_4^{2-}) form, and it can be blended with other dry fertilizer granules but SO_4^{2-} is prone to leaching. Recently, fertilizer industries introduced micronized S (MS) fertilizer formulations in the hopes that the smaller elemental S particles would increase the rate of S oxidation. Across the Red River Valley of North Dakota and Minnesota, field trials were conducted to compare the response of corn (*Zea mays* L.), spring wheat (*Triticum aestivum* L.), and sugar beet (*Beta vulgaris* L.) to application of different forms of S [(AS, ATS, monoammonium phosphate MAP-10S (5%ES + 5%AS), MAP +MS, AS +MS, muriate of potash (MOP) +MS, urea ammonium nitrate (UAN) +MS]. Spring wheat only had a significant positive response to S forms, with ATS having the highest grain yield in 2019; significantly higher than AS and AS + MS. In 2020, UAN + MS had the highest grain yield, only higher than control. Corn and sugar beet did not respond to S forms. Corn, sugarbeet, and spring wheat were indifferent to supply and forms of S.

Introduction

Crop nutrient management plays a critical role in optimizing production, and S is ranked just after N, P, and K (Amanullah & Fahad, 2018; Fageria et al., 2008; Stewart & Roberts, 2012). Sulfur deficiency is common in areas with very deep, low-organic-matter, sandy-textured soils (Rehm, 2005), but S deficiency symptoms are increasingly appearing outside of this area due to low atmospheric deposition (Franzen, 2015) and low S impurities in P fertilizers (Scherer, 2001). In the North-Central region, the standard soil test method to determine the soil S availability

(mono-calcium phosphate extraction) cannot predict the crops' response to S (Franzen, 2018). Several soils profiles contain high amount of native gypsum (CaSO_4) in subsoil (>60 cm). The complexation of Ca^{2+} ions in the native gypsum with extracted SO_4^{2-} results in inaccurate available SO_4^{2-} concentration. Due to high evapotranspiration demand in summer, dissolved SO_4^{2-} of gypsum moves upward and becomes available to plants. With spring snowmelt, SO_4^{2-} can leach out from the surface reducing the availability of S to plants (Carciochi et al., 2019). In a sandy loam soil, (Riley et al., 2002) determined that 72% of fertilizers were leached, and none of the fertilizer S from the ammonium sulfate [AS; $(\text{NH}_4)_2\text{SO}_4$] source remained in the root zone.

The most widely used sulfur (S) fertilizer sources in the northern Great Plains are ammonium sulfate (AS) [$(\text{NH}_4)_2\text{SO}_4$], ammonium thiosulfate (ATS) [$(\text{NH}_4)_2\text{S}_2\text{O}_3$], and elemental sulfur (ES). Plants absorb S primarily through the roots from the soil solution as sulfate-S ($\text{SO}_4\text{-S}$). Before S can be utilized by the plant, it must be converted to sulfate (SO_4^{2-}). The thiosulfate ion ($\text{S}_2\text{O}_3^{2-}$) in ATS rapidly oxidizes in soil to plant-available SO_4^{2-} . Ammonium sulfate applications can be made in spring and are available for crop uptake upon dissolution and movement into soil solution in the rhizosphere.

The cost of elemental S fertilizers is generally lower than $\text{SO}_4\text{-S}$ fertilizers (Grant et al., 2012). Oxidation of ES to SO_4^{2-} is mediated by soil microorganisms, including chemolithotrophs, such as *Thiobacillus* and, photoautotrophs, which consist largely of purple and green S bacteria, and heterotrophs which include a wide range of bacteria and fungi (Germida & Janzen, 1993). Factors controlling microbial activity also influence the rate of S oxidation. Application of ES is sometimes preferred over $\text{SO}_4\text{-S}$ in the belief that its slow oxidation to SO_4^{2-} might reduce leaching losses of SO_4^{2-} , however, this slow oxidation rate may also reduce available S to the crop at critical stages of growth. Reduction in particle size may increase the

oxidation rate. Availability of ES may be enhanced by formulations designed to reduce the particle size and increase the surface area exposed to microbial activity. Therefore, the modification of the particle size of ES is the most powerful tool in accelerating the oxidation rate. The success of combined S sources (ES + AS) products under field conditions depend on (i) oxidation rate of ES, (ii) chance of leaching for AS-S, (iii) ratio of ES and AS, and (iv) other soil and environmental conditions. Chien et al. (2016) concluded that the granular form of ES, in most cases, may not benefit crops planted after its application due to the ‘locality effect’ of $\text{SO}_4\text{-S}$ after ES oxidation due to the localized placement of ES particles around the applied granule sites after incorporation.

Evaluation of micronized ($<100\ \mu\text{m}$) S (MS) applications is necessary to justify its relevance over the more common AS, ATS, and ES. Degryse et al. (2020) found that ES might be more suitable than $\text{SO}_4\text{-S}$ in warm, humid climates; however, $\text{SO}_4\text{-S}$ should be recommended in colder climates as slow oxidation limits the initial availability of ES.

Research objectives are (i) to determine the S response of corn (*Zea mays* L.), wheat (*Triticum aestivum*), and sugar beet (*Beta vulgaris*), (ii) compare different forms of S in terms of grain yield and plant-S concentrations in North Dakota. We hypothesized that the application of S would increase the crops yield, and micronized forms of S would be supply more available S compared to $\text{SO}_4\text{-S}$ forms in wet seasons because SO_4^{2-} is more prone to leaching.

Materials and methods

The experiment design for all three crops was a randomized complete block design with eight treatments and four replicates. The eight treatments were (i) control (no-S), (ii) ammonium sulfate (AS), (iii) ammonium thiosulfate (ATS), (iv) MicroEssentials [Nutrien AG solution; MAP-10S (5%ES + 5%AS)], (v) MAP + MS, (vi) AS + MS, (vii) MOP + MS, (viii) UAN + MS.

Sulfur treatments and their respective N, P, K, and S concentration were given in Table 10. Sulfur application for all eight treatments was adjusted to 17 kg ha⁻¹. Nitrogen (N), phosphorus (P), and potassium (K) were applied according to the North Dakota fertilizer recommendations at the time of the experiments (Franzen, 2018). The N fertilizer application rate was balanced in each experimental unit with consideration of the residual soil nitrate to a 60cm depth determined before planting (within 14 days). All fertilizers were applied immediately before planting and incorporated into the soil using a field cultivator. Initial soil properties and fertilizer rates are provided in Table 11. Soil samples were collected before fertilizer application from depths of 0-15 and 15-60 cm, air dried, and ground to pass through a 2-mm sieve. The 0-15cm soil samples were analyzed for pH (1:1 soil/water) (Watson & Brown, 1998), electrical conductivity (Whitney, 1998), soil organic matter by loss on ignition at 360°C (Combs & Nathan, 1998), Olsen-phosphorus (P) (Frank et al., 1998), potassium (K) using the 1-N ammonium acetate extraction (Warncke & Brown, 1998) and sulfate-S (SO₄-S) using the mono-calcium phosphate extraction method and BaCl₂ turbidity (Combs et al, 1998). Soil samples of both depths were analyzed for nitrate-nitrogen (NO₃-N) using potassium chloride extraction and autoanalyzer (Gelderman & Beegle, 1998). Rainfall data were given in Table 12. Weather data was collected from nearby the North Dakota Agricultural Weather Network (NDAWN, <http://ndawn.ndsu.nodak.edu/>).

Corn

Field trials for corn were conducted at Ada (MN) and Chaffee (ND) during the 2019 and 2020 growing seasons, respectively. The experimental unit length and width were 7.60 m and 3.35 m, respectively. The inter-row spacing was 0.56 m with six rows within the experimental unit at both sites. The hybrid DKC35-88RIB was planted at a seeding rate of 85,000 plants ha⁻¹.

Five random plants per experimental unit were collected during 2019-2020 from each experimental site at the V6 growth stage and during the harvesting of corn. Plants were obtained from rows not intended for harvest. Plant samples were dried at 60° C and then ground to pass through a 2-mm sieve. One-half gram of plant material was digested with 20 ml of concentrated nitric acid (Soltanpour & Havlin, 1980) and analyzed for S concentration using inductively coupled plasma emission spectroscopy.

Table 10. Abbreviation used and N, P, K and S concentration of eight treatments used to test corn, sugar beet, and spring wheat response to S forms.

Sulfur treatments	Abbreviations	% wt.			
		N	P ₂ O ₅	K ₂ O	S
No sulfur, only N, P, and K	Control	-	-	-	-
Ammonium sulfate	AS	21	0	0	24
Ammonium thiosulfate	ATS	12	0	0	26
MicroEssentials	MES-10	12	40	0	10
Monoammonium phosphate + Micronized S	MAP + MS	9	43	0	16
Ammonium sulfate + Micronized S	AS + MS	17	0	0	36
Muriate of potash + Micronized S	MOP + MS	0	0	50	15
Urea ammonium nitrate + Micronized S	UAN + MS	5	0	0	38

The middle two rows from each experimental unit were hand-harvested to estimate the yield for all the sites during the 2019-2020 growing seasons. Grain moisture and test weight were measured using Dickey John Grain Moisture tester (GAC 500 XT, Illinois, USA). Grains were dried at 60° C and adjusting grain yield to 155 g kg⁻¹ moisture. The yield was calculated using the following equation.

$$\text{Yield (Mg ha}^{-1}\text{)} = [\text{Harvested grain wt. (Mg)} \times (10,000 \text{ m}^2 / \text{length of the row (m)} \times \text{width of the row (m)}) \times [(100 - \text{moisture}) / (100 - 0.155)]] \quad (\text{Equation 3})$$

Table 11. Location, soil properties and fertilizer application rates of experimental sites for three crops during 2019 and 2020 growing seasons.

Characteristics	Corn		Sugar beet		Spring wheat	
	2019	2020	2019	2020	2019	2020
Site	Ada, MN	Chaffee, ND	Ada, MN	Ada, MN	Wheatland, ND	Casselton, ND
Previous crop	Spring wheat	Sugar beet	Spring wheat	Spring wheat	Soybean	Soybean
Soil Series	Augsburg	Glyndon	Augsburg	Augsburg	Bearden	Bearden
Texture	Sandy clay loam	Clay loam	Sandy clay loam	loam	Clay loam	Clay loam
P (mg kg ⁻¹)	8	6	8	7	31	4
K (mg kg ⁻¹)	93	96	93	95	704	289
pH (1:1)	7.6	8.6	7.6	8.2	7.8	7.7
EC ^a (dS/m)	1.19	0.30	1.19	0.36	0.64	0.32
Organic matter (g/kg)	31	12	31	30	66	51
NO ₃ -N (kg/ha)	16.1	22	16.1	37.3	12	13
SO ₄ -S (kg/ha)	15	22	15	32	41	32
Planting date	8-May	26-Apr	13-May	11-May	26-Apr	2-May
Harvesting date	8-Oct	14-Oct	16-Sep	17-Sep	6-Aug	4-Aug
N (kg N ha ⁻¹)	250	250	146	146	168	280
P (kg P ₂ O ₅ ha ⁻¹)	58	117	52	62	17	67
K (kg K ₂ O ha ⁻¹)	101	101	101	101	0	0

^aEC, electrical conductivity

Sugar beet

Field trials with sugar beet were conducted at Ada, MN, during the 2019 and 2020 growing seasons. Individual treatment experimental units were measured 3.35-m wide and 9.14-m long. Each experimental unit contained six sugar beet rows spaced 55.9-cm apart. Crystal 093, a glyphosate-tolerant sugar beet cultivar, was planted at a rate of 148,200 plants per ha⁻¹. Sugar beet seed was planted to a 5-cm depth with a six-row John Deere Maximerge™ planter (John Deere, Moline, IL). A sample of 15-20 petioles of sugar beet was collected during 2019-2020 from the first leaf fully grown from the center of the whorl.

Sugar beets were mechanically defoliated at the time of sugar beet harvest. The top of the outside beets adjacent to the alleyways in each harvest row were spray painted to avoid including these sugar beets in the sugar analysis subsample. A scale-mounted sugar beet root harvester was used to dig and weigh the sugar beet roots from the center two rows of each plot. A sample of 15-20 petioles of sugar beet at harvest date was analyzed to determine S concentration using inductively coupled plasma emission spectroscopy (ICP) (Thermo Scientific-ICAP 6500, Waltham, MA, USA) after nitric acid digestion (Soltanpour & Havlin, 1980). A sub-sample of sugar beet roots was analyzed to determine sucrose concentration and recoverable sucrose at American Crystal Sugar Quality Tare Lab, East Grand Forks, MN. Recoverable sucrose yield was calculated using Carruthers et al. (1962a, 1962b) equation as modified by American Crystal Sugar Co. (Moorhead, MN) to calculate payments to individual growers (Campbell & Fugate, 2015).

$$\text{Recoverable sucrose yield (RSY) (Mg ha}^{-1}\text{)} = [\text{yield (Mg ha}^{-1}\text{)} \times (\text{sucrose \%} - \text{sucrose lost to molasses (SLM) \%}) / 100]$$

(Equation 4)

Spring wheat

Field trials for spring wheat were conducted at Wheatland, ND and Casselton, ND during the 2019 and 2020 growing seasons, respectively. In 2019 and 2020, the experimental unit length and width were 7.60 m and 3.35 m, respectively. In-season, twenty flag leaf samples per experimental unit were obtained during 2019-2020 at Feeke 11 growth stage from each experimental unit. Flag leaf samples were dried at 60°C, ground to pass through a 2-mm sieve, digested in concentrated nitric acid and analyzed for S concentration using inductively coupled plasma emission spectroscopy (ICP) (Thermo Scientific-ICAP 6500, Waltham, MA, USA) after nitric acid digestion (Soltanpour & Havlin, 1980). At physiological maturity, spring wheat grain yield was determined by harvesting each experimental unit using an Almaco™ (Almaco, Inc., Nevada, IA, USA) plot combine. Grain moisture content and test weight were measured using Dickey John Grain Moisture tester (GAC 500 XT, Illinois, USA). Grain yield is reported at 130 g kg⁻¹ moisture content. A subsample of grain was collected from each experimental unit at harvest. The samples were dried at 60 °C, ground using a Perten flour mill, and analyzed for protein concentration by near-infrared spectroscopy diode array (Perten Instruments, Stockholm, Sweden).

Statistical analyses

An overall ANOVA was performed using the PROC MIXED procedure in SAS for evaluating growing year, treatment (S rate) and their interaction effects on grain yield of corn, root yield of sugar beet and grain yield of spring wheat, S concentration, and other parameters related to corn, sugar beet and spring wheat. Significant differences were determined at 0.05 level using an LSD test. Sulfur forms and year were considered as fixed effects, and block (replication) was considered as a random effect.

Results

Growing season condition

Total monthly precipitation for all three crops during the 2019-2020 growing seasons are presented in Table 12. Considering all the experimental sites for the three crops (corn, sugar beet, and spring wheat), the cumulative rainfall from May through October was higher in 2019 as compared to 2020. In the case of all experimental sites for the three crops in 2019, all months (May-Oct) received more rainfall than the 30-yr normal (1981-2010) except for May and June. In 2020, all months (May-Oct) received less rainfall than the 30-yr normal except for July and August.

Corn

Year, S treatments, and their interaction had no significant effect on grain yield, tissue S concentration at V6 stage, and grain S uptake. The S treatments and the interaction of growing season and S treatments had no effect on grain yield or tissue S concentration at V6; however, the grain S uptake was greater in 2020 than in 2019 (Table 13).

Sugar beet

Growing season, S treatments, and their interaction effects on sugar beet root yield, petiole S concentration, and sugar concentration are provided in Table 14. Only year had a significant effect on root yield with 2019 having a higher root yield than 2020.

Spring wheat

Sulfur application increased wheat grain yield compared with the control in 2020, but not in 2019 (Table 6). Mean grain yield was greater in 2020 than in 2019. In 2019, the application of ATS had the highest yield (5.08 Mg ha^{-1}), significantly higher than the control (4.73 Mg ha^{-1}). The lowest yield was observed with the application of AS + MS (3.67 Mg ha^{-1}). In 2020, yields

with all S treatments were greater than that of the control. Considering both seasons, ATS had the highest grain yield (5.26 Mg ha^{-1}) compared with other treatments. Sulfur forms had no significant effect on grain sulfur concentration and protein concentration. Only year had a significant effect on grain S concentration and protein concentration. Considering both years, S concentration was similar among treatments. Grain protein concentration declined in 2020 compared with 2019. Grain protein was similar in response to different S forms.

Discussion

Six field trials with seven forms of S and three different crops over 2 yr found that year had more pronounced effect on yield parameters than S addition and S forms. Spring wheat and sugar beet trials were responsive to year, but not corn. Mean grain yield of spring wheat was greater in 2020 than in 2019; in contrast, the sugar beet root yield was higher in 2019 than in 2020. In 2019, lower-than-average May and June rainfall reduced spring wheat grain yield, but extremely high rainfall in fall 2019 might facilitate the grain yield for the following year. Sugar beet was planted almost 2 weeks after spring wheat, and harvested in September, so sugar beet root yield might be negatively influenced by reduced overall growing season (May–October) rainfall in 2020. A lack of response to S and suggested that S from SOM and its mineralization in some soils was enough for crop growth (Kaur et al., 2019). For the spring wheat site, SOM concentration was 66 and 51 g kg^{-1} for 2019 and 2020, respectively (Table 2). For corn and sugar beet, however, SOM concentration was $\leq 31 \text{ g kg}^{-1}$. Moreover, sites under spring wheat had clay loam soils. In Michigan, Steinke et al. (2015) found that relatively high SOM ($>28 \text{ g kg}^{-1}$) and residual S ($>6\text{--}8 \text{ mg kg}^{-1}$) is enough to achieve maximum corn yield without application of S. In the northern Great Plains, S deficiency is controlled by soil properties

Table 12. Total rainfall average and departure from normal (1981-2010, DNa) for the experimental sites.

Month	Corn				Sugar beet				Spring wheat			
	2019		2020		2019		2020		2019		2020	
	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN	Avg.	DN
	mm											
May	62.4	-19.9	22.1	-54.6	62.4	-19.9	33.8	-48.5	60.0	-17.5	41.1	-36.3
June	68.3	-45.5	75.7	-17.0	68.3	-45.5	57.2	-56.6	122	-21.7	79.0	-21.3
July	103	9.78	160	78.3	103	9.78	102	9.50	156	68.2	122	34.9
August	93.7	24.1	115	52.9	93.7	24.1	15	89.0	102	35.9	115	49.1
September	106	38.9	16.5	-47.0	106	38.9	16.0	-51.1	147	82.1	13.2	-52.3
October	92.2	35.3	9.90	-43.4	92.2	35.3	10.9	-46.0	77.0	15.3	7.6	-54.1
Total	525	42.3	401	-30.8	525	42.3	379	-103	664	205	378	-80.0

^aDeparture from normal was calculated by the deviation from the 30-yr (1981-2010) average val

Table 13. Corn grain yield, tissue sulfur concentration at V6 stage and grain sulfur uptake affected by different sulfur forms during 2019 and 2020 growing seasons.

Treatment	Grain yield			Tissue S conc. at V6 stage			Grain S uptake		
	2019	2020	Mean	2019	2020	Mean	2019	2020	Mean
	Mg ha ⁻¹			g kg ⁻¹			kg ha ⁻¹		
Control	13.3 (0.32)	13.7 (0.93)	13.5 (0.63)	3.09 (0.27)	3.30 (0.18)	3.20 (0.23)	11.1 (0.27)	14.0 (1.23)	12.6 (0.75)
AS	12.4 (0.60)	13.4 (1.42)	12.9 (1.01)	3.61 (0.29)	3.23 (0.35)	3.42 (0.32)	10.6 (0.89)	14.0 (1.62)	12.4 (1.26)
ATS	13.4 (0.94)	12.6 (3.33)	13.0 (2.14)	3.28 (0.27)	3.15 (0.44)	3.22 (0.36)	12.1 (1.58)	13.0 (3.86)	12.6 (2.72)
MES-10	12.7 (0.37)	14.5 (1.45)	13.7 (0.91)	3.31 (0.13)	3.05 (0.79)	3.18 (0.46)	10.8 (0.40)	14.2 (1.38)	12.5 (0.89)
MAP + MS	12.4 (0.89)	13.2 (3.39)	12.9 (2.14)	3.34 (0.38)	3.20 (0.50)	3.27 (0.44)	10.8 (0.40)	13.8 (3.98)	12.4 (2.19)
AS + MS	13.0 (0.72)	13.3 (2.67)	13.2 (1.70)	3.44 (0.47)	3.35 (0.35)	3.40 (0.65)	11.6 (0.77)	13.8 (3.66)	12.7 (2.22)
MOP + MS	13.1 (0.47)	13.0 (1.03)	13.1 (0.75)	3.20 (0.39)	3.00 (0.42)	3.10 (0.41)	11.4 (0.81)	13.1 (1.94)	12.3 (1.38)
UAN + MS	12.8 (1.39)	14.3 (1.23)	13.6 (1.31)	3.33 (0.28)	3.00 (0.22)	3.17 (0.25)	10.8 (1.31)	14.9 (1.11)	12.9 (1.21)
Mean	12.9 (0.71)	13.5 (1.93)	13.2 (1.32)	3.32 (0.31)	3.16 (0.41)	3.24 (0.36)	11.1 (0.80) a [†]	13.8 (2.35) b	12.5 (0.58)
ANOVA									
Year	NS			NS			<0.001*		
Treatment	NS			NS			NS		
Year x Treatment	NS			NS			NS		

Note. Values in parentheses indicate the standard deviation.

*Significant at 0.05 probability level; NS = non-significant

[†] Means with different letters were determined to be significantly different at $P < 0.05$ using the LSD test

Table 14. Sugar beet root yield, petiole sulfur concentration and sugar concentration affected by different sulfur forms during 2019 and 2020 growing seasons.

Treatment	Root yield			S concentration			Sugar Concentration		
	2019	2020	Mean	2019	2020	Mean	2019	2020	Mean
	Mg ha ⁻¹			g kg ⁻¹			%		
Control	72.2 (1.81)	60.3 (6.21)	66.2 (4.01)	2.58 (0.24)	2.72 (0.25)	2.65 (0.25)	17.0 (0.36)	17.7 (0.27)	17.4 (0.32)
AS	71.7 (5.73)	65.5 (3.47)	68.6 (4.60)	2.68 (0.53)	2.80 (0.24)	2.74 (0.39)	17.0 (0.32)	18.0 (0.49)	17.5 (0.41)
ATS	73.3 (1.54)	59.8 (6.05)	66.6 (3.80)	2.54 (0.25)	2.55 (0.25)	2.55 (0.25)	16.8 (0.36)	17.5 (0.64)	17.2 (0.50)
MES-10	73.8 (2.64)	66.7 (4.80)	70.3 (3.72)	2.64 (0.40)	2.87 (0.10)	2.67 (0.25)	16.8 (0.46)	18.0 (0.23)	17.4 (0.35)
MAP + MS	69.5 (1.74)	62.8 (7.58)	66.2 (4.66)	2.51 (0.13)	2.70 (0.08)	2.61 (0.11)	17.2 (0.41)	17.8 (0.40)	17.6 (0.41)
AS + MS	73.3 (2.29)	66.5 (4.09)	69.9 (3.19)	2.63 (0.22)	2.72 (0.10)	2.68 (0.16)	16.9 (0.42)	17.7 (0.39)	17.3 (0.41)
MOP + MS	70.1 (0.78)	65.1 (3.56)	67.6 (2.17)	2.42 (0.08)	2.62 (0.22)	1.52 (0.15)	17.1 (0.23)	17.5 (0.22)	17.3 (0.23)
UAN + MS	71.3 (4.18)	64.3 (7.61)	67.8 (5.90)	2.59 (0.29)	2.55 (0.42)	2.57 (0.36)	16.6 (0.60)	17.8 (0.67)	17.2 (0.64)
Mean	72.3 (2.59) b [†]	61.2 (5.42) a	66.8 (5.30)	2.57 (0.27) a [†]	2.69 (0.21) b	2.63 (0.24)	16.9 (0.40) a [†]	17.7 (0.41) b	17.4 (0.41)
ANOVA									
Year	<0.001*			<0.04*			<0.001*		
Treatment	NS			NS			NS		
Year x Treatment	NS			NS			NS		

Note. Values in parentheses indicate the standard deviation.

*Significant at 0.05 probability level; NS = non-significant

[†] Means with different letters were determined to be significantly different at $P < 0.05$ using the LSD test

Table 15. Spring wheat grain yield, S concentration and protein content affected by different sulfur forms during 2019 and 2020 growing seasons.

Treatment	Grain yield			S concentration			Protein content		
	2019	2020	Mean	2019	2020	Mean	2019	2020	Mean
	Mg ha ⁻¹			g kg ⁻¹					
Control	4.73 (0.27) bc	5.08 (0.16) a	4.90 (0.22) b	3.13 (0.12)	2.03 (0.28)	2.58 (0.20)	166 (7.54)	155 (3.63)	161 (5.89)
AS	4.44 (0.43) b	5.20 (0.21) ab	4.97 (0.32) b	3.17 (0.24)	1.95 (0.57)	2.56 (0.41)	164 (6.26)	152 (5.58)	158 (5.92)
ATS	5.08 (0.77) c	5.44 (0.38) ab	5.26 (0.58) c	3.10 (0.19)	1.93 (0.29)	2.52 (0.24)	161 (6.96)	148 (6.77)	155 (6.87)
MES-10	4.64 (1.03) bc	5.33 (0.26) ab	4.99 (0.65) bc	3.10 (0.28)	2.05 (0.40)	2.58 (0.34)	162 (6.13)	149 (5.58)	156 (5.86)
MAP + MS	5.02 (0.95) c	5.13 (0.42) ab	5.08 (0.69) bc	2.92 (0.20)	2.05 (0.29)	2.49 (0.25)	164 (4.57)	147 (2.26)	156 (3.42)
AS + MS	3.67 (0.61) a	5.23 (0.60) ab	4.45 (0.51) a	2.93 (0.33)	2.08 (0.15)	2.51 (0.24)	164 (7.00)	153 (7.76)	159 (7.38)
MOP + MS	4.63 (0.89) bc	5.26 (0.43) ab	4.95 (0.66) bc	2.92 (0.43)	2.08 (0.34)	2.50 (0.39)	164 (7.88)	151 (6.39)	158 (7.14)
UAN + MS	4.67 (0.54) bc	5.55 (0.48) b	5.11 (0.51) bc	2.99 (0.14)	1.93 (0.46)	2.46 (0.30)	163 (8.03)	149 (4.02)	156 (6.03)
Mean	4.65 (0.69) a [†]	5.21 (0.37) b	4.93 (0.53)	3.00 (0.20) b [†]	2.89 (0.35) a	2.95 (0.28)	164 (6.80) b	151 (5.25) a	158 (6.03)
ANOVA									
Year	<0.0001*			<0.001*			<0.001*		
Treatment	0.001*			NS			NS		
Year x Treatment	0.003*			NS			NS		

Note. Values in parentheses indicate the standard deviation.

*Significant at 0.05 probability level; NS = non-significant

[†] Means with different letters were determined to be significantly different at $P < 0.05$ using the LSD test.

(Franzen & Grant, 2008). In the North-Central region, soils contain gypsum deeper (>30 cm) in the soil profile particularly for Fargo (fine, smectitic, frigid Typic Epiaquerts) and Bearden (fine-silty, mixed, superactive, frigid Aeric Calciaquolls) soil series (USDANRCS Web Soil Survey, <https://websoilsurvey.sc.egov.usda.gov/>). After spring snow melt, SO_4^{2-} leach down in sandier soils, contributing to generally less available S. During the summer season, due to high evapotranspiration demand, dissolved gypsum ($\text{SO}_4\text{-S}$) from the subsurface of soil moves upward with soil water, and S becomes available for uptake by plants (Nachshon et al., 2013). Among S forms, spring wheat showed inconsistent response. In these studies, crop responses to S fertilization were not found in 2019 and 2020 in sugar beet and corn; however, there were yield increases to S fertilization in both years in spring wheat. There was little difference in crop S response to different forms of S. This study indicated that elemental S, applied as micronized S, may be as effective as SO_4^{2-} and $\text{S}_2\text{O}_3^{2-}$ forms. The superiority of ATS in the wetter growing season of 2019 did not appear to be related to S but may have been due to its effect at slowing nitrification and limiting denitrification (Goos & Johnson, 1992, 1999). Denitrification loss of N is generally high in clay loam soils with high SOM concentration (Chatterjee, 2020). Goos and Johnson (1992, 1999) observed that ATS had value as a nitrification inhibitor and increased spring wheat grain yield in a wet spring in Fargo silty clay soils. Commercial batches of UAN sometimes contain ATS (added to reduce crystallization temperature) and might affect the ATS alone (Goos, 1985).

Conclusion

Corn, spring wheat, and sugar beet yields were more responsive to interaction between growing season and soil characteristics rather than addition of S and S forms. Mineralization of S and S present in deep soil profile may be enough to fulfill the crops' S demand. Lack of

variations among S forms indicate that they are similar in terms of supply of S. For corn, growers should apply the current recommendation of 11–22 kg S ha⁻¹ to reduce the chance of yield loss and compensate for the removal with grain. No additional response of S addition can be expected for spring wheat and sugar beet.

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GENERAL CONCLUSION

This study showed the corn response to S application over multiple sites and growing seasons. Corn grain yield is affected by rainfall and crop residue of previous crop. Across three growing years, corn grain yield was significantly greater in 2019 (15 Mg ha⁻¹) than in 2018 (14.5 Mg ha⁻¹) and 2020 (12.8 Mg ha⁻¹). As rainfall is higher in 2019 (519 mm) than in 2018 (433 mm) and 2020 (390 mm). Across 12 site-year, Walcott II in 2019 had the greatest average site yield (19.8 Mg ha⁻¹). This site received the maximum rainfall (633 mm) along with previous crop residue of soybean crop. Soybean is a leguminous crop, and its residue is a good source of nitrogen. This study showed that corn yield and S uptake did not influence by S additions irrespective of soil type over three growing seasons. It is also evident that neither SOM nor SO₄-S soil test could predict the corn response to S.

Corn, sugar beet and spring wheat responses to different forms of S was also studied. Growing season and different S treatments had no impact on corn and sugar beet crop. There is no differences among the various S forms. However, there were yield increases to S fertilization in both years in spring wheat but had no impact on S concentration and protein content. In 2019, there was significantly increase in spring wheat yield with the application of ATS. In 2020, significant increase in yield was observed with the application of UAN along with MS. The grain yield increased not because of supply of S but ATS acted as nitrification inhibitor extending N availability later into growing season. Crop residue can also put an impact on increasing grain yield. In this study spring wheat sites in both seasons had crop residue of soybean crop. Nitrogen available from soybean crop residue can also help in increasing grain yield.

Overall, these studies suggest that application of S had no impact on corn, spring wheat and sugar beet crop but still S fertilizer should be applied at a rate of 11-22 kg S ha⁻¹ to reduce

the chance of yield loss due to removal of S by grain. Also in future studies, it will be more important to identifying the site characteristics to predict the yield response.

APPENDIX: SULFUR DEFICIENCY PICTURE



Figure A1. Sulfur deficiency at Chaffee II (site 11) in 2020 growing season.