CORN RESPONSE TO SULFUR FERTILIZER IN THE RED RIVER VALLEY

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The Supervisory Committee certifies that this ***disquisition*** complies with North Dakota State University's regulations and meets the accepted standards for the degree of

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ABSTRACT

A study was conducted at ten locations in North Dakota and Minnesota in 2016 and 2017 to evaluate corn response to different sulfur (S) application rates and to determine the relationship between corn yield and plant tests. Five S treatments of 0 (check), 11, 22, 33, and 44 kg S ha^{-1} were applied as ammonium sulfate granular fertilizer. Significant increase in corn yield occurred at only two sites (out of ten sites) in both year. Application of 33 kg S ha^{-1} (2016) and 44 kg S ha^{-1} (2017) increased corn yield by 3.4 Mg ha^{-1} and 1.3 Mg ha^{-1}, respectively. Poor correlation was noticed between plant tests (tissue S and N/S) and corn yield. These results indicate that response to S varies from soil to soil and weather conditions may play the most important role in determining the response. Additional research should be conducted using different soils over multiple years.

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1. LITERATURE REVIEW

1.1. Sulfur in plants

Sulfur is an essential element required for normal plant growth, a fact that has been recognized since 1860 (Alway, 1940). It is considered a secondary macronutrient, following the primary macronutrients nitrogen, phosphorus, and potassium, but is needed by plants at levels comparable to P (Kovar and Grant, 2011). Plants require S for synthesis of cystine, cysteine and methionine, which are amino acids that form an integral part of proteins (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 nitrogen fixation (Brady and 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 and 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, SO_4^{2-} (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 and 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. Apart from crop yield, S deficiency can also affect crop quality. Haneklaus et al., (1992) found that insufficient S diminished the baking quality of wheat (*Triticum aestivum* L.) well before crop productivity decreased. Defects in potato (*Solanum tuberosum* L.) tubers often result when S uptake is below optimum. Pavlista, (2005) found that common scab

(*Streptomyces scabiei*) and black scurf (*Rhizoctonia solani*) were reduced by early-season applications of elemental S, ammonium sulfate, or ammonium thiosulfate during a 6-yr study in the western United States. Haneklaus et al., (2008) concluded that a balanced nutrient supply, including S fertilization, for agricultural crops is the best guarantee for producing healthy foods.

1.2. Sulfur in soils

Total S in soil varies widely depending on soil organic matter content, parent material, fertilizer amendments and atmospheric depositions (Scherer, 2009). Soil S concentration can range from 20 mg kg^{-1} in highly weathered humid soils to 50,000 mg kg^{-1} in calcareous and saline soils of arid and semi-arid region (Stevenson, 1986). Generally, the soil S pool is divided into two major groups: organic and inorganic S. Around 95-98% of total soil S exists in the organic form (Rehm and 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 to the mobile sulfate form (SO_4^{2-}) that is available for plant uptake (Scherer, 2001).

Organic S forms are further divided into two main groups: ester sulfates and carbon bonded S; with C-O-SO_3 linkages in the former and direct C-S linkage in the latter. Generally, ester sulfates are derived from microbial biomass material (David et al., 1984; Mclaren et al., 1985) whereas C-S is formed from plant residue (Konova, 1975; David et al., 1984). Choline sulfates, sulfated polysaccharides and phenolic sulfates are included in theester sulfate class (Edwards, 1998); whereas the S-containing amino acids and sulpho-lipids (Tabatabai and Bremner, 1972; Neptune et al., 1975; Harwood and Nicholls, 1979) are included in the C-S class. These two groups can be differentiated using laboratory fractionation techniques proposed by Johnson and Nishita, (1952), Freney et al., (1970), Landers et al., (1983) and Shan and Chen, (1995) in which ester sulfate is determined using hydriodic acid (HI) reduction and C-S from the difference between total organic S and ester sulfate. Ester sulfates provide S more rapidly under microbial mineralization (Fitzgerald, 1978; Strickland et al., 1987) than C-S (Hutchinson, 1979; Schindler et al., 1986). Ester sulfates serve as only a temporary soil reserve of S as with time it is converted into C-S.

Compared to the organic S pool, inorganic S pool is less abundant in soil (Bohn et al., 1986). It includes sulfate (SO_4^{2-}), sulfide (S^{2-}), sulfite (SO_3^{2-}) and elemental S (S^0) (Tabatabai, 1996). Sulfate is the dominant form in most soil. It can be present as SO_4^{2-} in soil solution, adsorbed on colloidal surfaces (Barber, 1995) or co-precipitated with calcium and magnesium (Tisdale et al., 1993). Sulfate in soil solution is in equilibrium with the solid S forms (Mengel and Kirkby, 1987; Tisdale et al., 1993). Soils composed in part as Fe and Al oxides in tropical soils can adsorp considerable sulfate on either Fe, Al coatings or edges of aluminosilicate clay particles (Bohn et al., 1986). Soil pH, silicate clay mineral, amorphous Fe and Al oxides and presence of other anions control sulfate adsorption. Adsorption is higher in low pH soils, being highest at pH 3 (Scherer, 2009) and negligible after pH 6.5 where most of sulfate is in soil solution (Curtin and Syers, 1990). On silicate clays, sulfate adsorption follows the order kaolinite > illite > montmorillonite. Among anions, phosphate anion gives highest competition for adsorption followed by nitrate and chloride (Tisdale et al., 1993) so application of phosphatic fertilizers can help in increasing S availability for plant uptake in soils with significant anion exchange capacity. Although adsorped sulfate is unavailable for plant uptake adsorption limits S leaching losses (Scherer, 2001).

1.3. Sulfur mineralization and immobilization

The organic S fractions in soil are unavailable for plant uptake until they are converted to inorganic-S through biochemical or microbiological mineralization (Castellano and Dick, 1991).

Biochemical mineralization involves the hydrolyzation of ester sulfates by various sulfatases (sulfohydrolase) (Fitzgerald and Strickland, 1987) while sulfide mineralization occurs when microbes use C-S as a C source and release sulfate as its oxidized by-product (Ghani et al., 1991). The microbial enzyme arylsulfatase helps in catalyzing the hydrolysis of sulfate esters. Ester sulfates are mineralized to satisfy microbial nutrition needs (for S) whereas C-bonded S mineralization is driven by the need to satisfy microbial energy needs (for C) (Edwards, 1998).

Temperature, moisture, organic matter, atmospheric deposition inputs, and other factors influence immobilization rates (Randlett et al., 1992, Freney et al., 1971). In cold, wet regions, temperature conditions are not conducive to microbial activity during the winter when substrates are available (Williams, 1967) which results in less S mineralization or immobilization. Swank et al., (1985) reported a slightly earlier peak for immobilization rates in August and September, with lowest rates in winter and late spring. Sulfur mineralization increased markedly with increasing temperatures at 10°, 20°, and 30°C over a 64-day incubation period (Williams, 1967).

The amount of S taken up by crops may be returned to the soil in their residues less the S contained in forage or grain removed. Incorporation of residues rich in S releases plant available S whereas residues poor in S can result in S immobilization (Scherer, 2001). Further, soil S status depends on soil organic matter content (Tabatabai and Bremner, 1972) and its C/S ratio. Generally, a C/S ratio of < 200:1 results in mineralization while a ratio > 400:1 results in immobilization of organic S compounds (Janzen and Kucey, 1988). For C/S ratios between 200 and 400, SO_4^{2-}can be either released from or tied up in the soil organic matter (Scherer, 2001).

1.4. Methods to determine sulfur

Soil testing procedures have been developed to measure the plant available S fractions including inorganic SO_4^{2-}, adsorbed SO_4^{2-} in soils with anion exchange capacity and S available from mineralization of organic S compounds (Bettany et al., 1974). One class of soil testing procedures involves the extraction of soil with a salt solution, followed by determination of extracted S. Several reagents have been utilized for extraction including $Ca(H_2PO_4)_2$ (Combs et al., 1998), KH_2PO_4 (Fox et al., 1964), Mehlich 3 (Mehlich, 1984), H_2O (Walker and Doomenbal, 1972) and salt solutions $CaCl_2$, NaCl or LiCl (Tabatabai, 1982). Among all, $Ca(H_2PO_4)_2$ is used most widely as phosphate ion can easily displace the adsorbed sulfate ions (Spencer and Freney, 1960; Williams and Steinbergs, 1964) better than other salts. After extraction, S can be determined using a turbidimetric method (Chesnin and Yien, 1962), a reduction-colorimetric method of Johnson and Nishita, (1952), or inductively coupled plasma-optical emission spectrometry (ICP-AES; Li et al., 2001).

There are several limitations of using a soil test as an indicator of S sufficiency and deficiency. Firstly, it determines the soil solution and adsorped sulfate only and does not consider organic S components that might become available after mineralization. Secondly, this method fails to work on gypsum rich soils (Spencer and Freney, 1960). The soils of Northern Great Plains may contain more than 5% gypsum that overestimates plant-available sulfate and interferes with turbidity development (Cihacek et al., 2015). Thus, soil testing is not diagnostic in these soils.

Plant analysis is accepted as a better tool in predicting S deficiency (Zhao et al., 1996). It involves the estimation of total plant S (Jones, 1986) and N/S ratio in plant material (Marschner, 1995). Total S concentration and N/S values are dependent on various factors like crop development stage, hybrid and growth conditions. For corn, optimum S concentration varies from 15 to 50 g kg^{-1} (Tandon, 1984) and N/S ratio as 16:1 (Stewart and Porter, 1969; Terman et al., 1973; Reneau, 1983). The major problem with plant analysis for diagnosis of S deficiency is that it indicates deficiency after it has occurred, which might be too late achieve maximum productivity from rescue S fertilizer application (Malhi et al., 2005).

1.5. Corn response to sulfur fertilizer

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 2015 (USDA, 2016). Similarly, average corn yield increased from 7.4 Mg ha^{-1} in 1985 to 10.6 Mg ha^{-1} in 2015 (USDA, 2016) because of high yielding varieties and efficient management practices.

Nutrient management plays a dominant role in increasing corn yield. Most of the management strategies focus on three essential nutrients namely, nitrogen, phosphorus and potassium. 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 SO_4-S via rainfall, fertilizer, and pesticides has decreased (Dick et al., 2008).

Many researchers in the US have reported corn response to S fertilizer where there has previously been yield increases in very sandy, low organic matter soils. Sawyer et al., (2009) observed an increase in corn yield at five of six Iowa sites with yield increase of 2.4 Mg ha^{-1} across all sites. This high yield was attributed to low soil S and severe S deficiency at the selected sites. Further, trials conducted in 2007-2008 showed a significant response of corn to S fertilizer at 17 of 20 sites in 2007 and 11 of 25 sites in 2008 (Sawyer et al., 2011). In the Atlantic Coastal Plains of the U.S., field research demonstrated that when residual soil S was equal to 1.5 mg kg^{-1} at planting the application of 22 and 44 kg S ha^{-1} increased corn yields from 1 to 28% (Reneau, 1983). In addition, there were certain studies showing inconsistent results to S application. In Nebraska, there was no response to applied S at all 11 sites years with different soil textures (Wortmann et al., 2009). A response was expected in sandy soils, but these soils had enough organic matter (10 mg kg^{-1}) to fulfill plant S demand.

In soybean, yield increase of approximately 134 kg ha^{-1} across S fertilizer rates of 0 to 56 kg S ha^{-1} were reported across various soil types in Minnesota in 2011 and 2012 (Kruger et al., 2014). The application of gypsum in Ohio in 2000, at rates of 16 kg S ha^{-1} and 67 kg S ha^{-1}, increased soybean yield by 4.8% and 11.6%, respectively (Chen et al., 2005). Similarly, in canola, the application of S in the form of flue gas desulfurization gypsum and N in the form of urea at rates of 33.6 kg S ha^{-1} and 30 kg N ha^{-1} increased yield (~50%) over N applied at a rate of 30 kg N ha^{-1} near Langdon, North Dakota (ND) (DeSutter et al., 2011). These studies demonstrated that, along with nitrogen, phosphorous and potassium, S is an important nutrient required for improving crop yield. Addition of S fertilizer in the fertilizer program might be needed for dealing with S deficiency in many North Dakota crops.

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2. CORN RESPONSE TO SULFUR FERTILIZER IN THE RED RIVER VALLEY

2.1. Abstract

The recent increase in incidence and severity of sulfur (S) deficiency in the North Dakota (ND) region requires revising current S recommendation for corn (*Zea mays*). The decrease in atmospheric S deposition in addition to large S removal by high yielding crop varieties has resulted in a reduction in growing season S availability. Ten on-farm field trials were conducted to evaluate the corn response to S additions in the Red River Valley of North Dakota and Minnesota in 2016 and 2017. Five S rates of 0, 11, 22, 33, 44 kg S ha^{-1} as granular ammonium sulfate were broadcast and incorporated prior to corn planting. The experimental design at each location was a randomized complete block with four replications. Plant tissue S concentration at the V6 and V12 stages was determined and corn grain yield was measured at harvest. Corn yield increased with increased S fertilizer rate at one site (out of 5) in 2016 and at one site (out of 5) in 2017. Yield increase was realized with the 33 kg S ha^{-1} rate at the one site in 2016, whereas in 2017, yield response was observed at 44 kg S ha^{-1} application rate. Plant tissue S and soil tests correlated poorly with corn yield. These results indicate that corn yield response to S varies with soil and weather conditions, and corn tissue S concentration is a poor predictor of grain yield response.

2.2. Introduction

Sulfur (S) is considered the fourth major nutrient for optimum plant growth (Franzen and Grant, 2008) and is required for several plant functions due to its role in the structure of certain amino acids and their protein products. As such, S has a prominent role in enzyme synthesis and activity and most metabolic processes in plants (Coleman, 1966). Unlike N, P and K in the central USA, researchers in this region have not studied S extensively mainly because S deficiency symptoms were seldom seen outside of very deep, low organic matter, sandy-textured soils. However, in recent years the incidence and severity of S deficiency has become widespread and common within many central states of the USA, including North Dakota and Minnesota (Eriksen et al., 2004; Girma et al., 2005). The higher frequency of S deficiency and its severity is related to higher crop yield, which imposes a greater demand on soil for available S, together with the reduction in atmospheric S deposition (Scherer, 2001) due to consequences of much stricter clean-air regulations and industry compliance in the USA and Canada. Across the USA, average atmospheric sulfur dioxide (SO_2) values in the air has been reduced by 87% from 1980 to 2016 (U.S. Environmental Protection Agency, 2017). In addition, use of high analysis phosphate with lower S impurities contribute to the lower plant-available S supply (Hagstrom, 1986; Chien et al., 2011).

Soil organic matter S reserves contribute S to crops annually through residue/organic matter decomposition and subsequent release of S (mineralization). Roughly, 95% of total soil S is present in organic form in many soils (Tabatabai, 1984), with the remaining 5% in inorganic form (SO_4^{2-}). Most S is taken up by plants in the sulfate (SO_4^{2-}) form. Reduced S, which is the product of S mineralization, is oxidized to sulfate through the activity of soil microorganisms (Dick et al., 2008). Higher S mineralization rates are related to soil organic matter concentration, temperature and favorable soil moisture (Schoenau and Malhi, 2008). Historically, S deficiency is most common in low organic matter coarse textured soils (Franzen and Grant, 2008). However, more recent research has reported an increasing incidence of S deficiency on medium and fine texture soils (Rehm, 2005; Sawyer et al., 2009, 2011; Franzen, 2015).

The average corn grain production in the USA has increased from 5.7 Mg ha^{-1} in 1980 to 10.9 Mg ha^{-1} in 2017 (USDA-NASS, 2017). Sulfur removed by corn grain ranges from 1.0 to 1.2

g kg^{-1} (Chen et al., 2008; Bender et al., 2013). Corn grain yield increases due to S application have been documented in many regions of the world (Weil and Mughogho, 2000; Prystupa et al., 2006; Chen et al., 2008; Pagani et al., 2009). In Iowa, 62% of randomly placed S-rate experiments in 2008 showed a grain increase with S application on coarse and fine textured soils (Sawyer et al., 2009), whereas before 2005 no corn S deficiency had ever been recorded in the state. In Minnesota, corn yield increased from 8.7 Mg ha^{-1} to 9.6 Mg ha^{-1} on sandy loam soils and from 9.3 Mg ha^{-1} to 10.1 Mg ha^{-1} on silt loam soils with S application (Rehm, 2005).

Soil analysis with the mono-calcium phosphate extraction method in the central states of the USA has previously been used as a standard method for recommending S applications (Hoeft et al., 1973). In addition to other problems associated with the failure to diagnose soil sulfur status in the region (Franzen, 2015), the presence of soil gypsum can also result in inaccurate results and faulty diagnosis (Spencer and Freney, 1960). Many soils in North Dakota contain high gypsum values. Due to the general poor performance of any sulfur soil test for predicting crop S response, plant analysis (tissue S and tissue N to S ratio) has been found more helpful in predicting S needs in season (Zhao et al., 1999).

According to current recommendation in ND, at least 11 kg S ha^{-1} should be applied if rainfall or snow-melt is above normal in low organic matter soils in fall, winter, or early spring (Franzen, 2018). This study was planned to revisit corn S fertilizer recommendation for the Red River Valley region. The objectives of this experiment were to determine corn response to S fertilizer application and to evaluate the relationship between corn yield and leaf tissue S analysis.

2.3. Material and methods

2.3.1. Research sites and soil analysis

Field S-rate experiments were established at ten experimental sites in North Dakota and Minnesota during 2016 and 2017 (Fig. 1). Six sites were located within corn producer fields and four were located on NDSU Research & Experiment Station land. All sites had been managed under a corn-soybean production system except for Ada I, which was in a corn-wheat cropping rotation. Soil samples were collected before fertilizer application to a depth of 15 cm, dried at 105º C, and ground to pass a 2-mm sieve. These samples were analyzed for pH (1:2 soil/water) (Watson and Brown, 1998), electrical conductivity (Whitney, 1998), soil particle size distribution (Gee and Bauder, 1986), soil organic matter by loss on ignition at 360ºC (Combs and Nathan, 1998), phosphorus using the Olsen method (Frank et al., 1998), potassium using the 1-N ammonium acetate extraction (Warncke and Brown, 1998) and sulfate-S using the mono-calcium phosphate extraction method and $BaCl_2$ turbidity (Combs et al., 1998). Soil samples were also obtained at the 0-60 cm and were extracted with water for NO_3-N analysis (Gelderman and Beegle, 1998). Initial soil characteristics are presented in Table 1. In-season soil samples were collected at V6 and R2 crop growth stages to the 30-cm depth and analyzed for S using the methods previously described.

Total precipitation and temperature data was collected from near NDAWN weather stations for all sites except sites 7, 8, and 10, where weather stations (Watchdog, Spectrum technologies) were installed to record precipitation and temperature data.

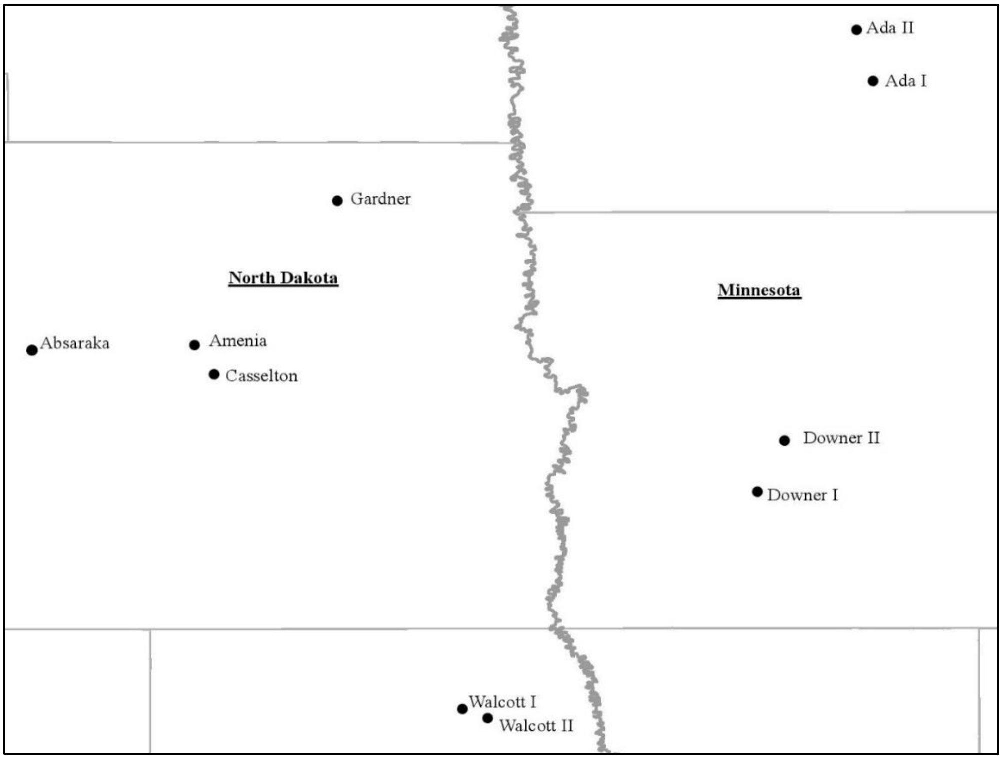


Fig. 1. Experimental sites selected in 2016-2017 growing season.

2.3.2. Experimental design

The experimental design was a randomized complete block with five S application rates (0, 11, 22, 33 and 44 kg S ha^{-1}) and four replications. Sulfur was applied as granular ammonium sulfate. The fertilizer was broadcasted and incorporated immediately before planting corn. In 2016, each experimental unit was 9.10 m by 3.35 m with an inter-row spacing of 0.56 m. Next year, the experimental unit length and width was 7.60 m and 3.35 m, respectively. The inter-row spacing was 0.56 m with 6 rows within the experimental unit at all sites except at Walcott II, where it was 0.76 m, with 4 rows within the experimental unit. Corn was planted at the seeding rate of 81,000 plants ha^{-1}. Roundup Max (Isopropylamine salt of glyphosate a.i.) 25 ml liter^{-1} was sprayed twice (last week of May and third week of June) to control weeds. At Downer, a mixture of RoundupMax 25 ml liter^{-1} and ClassAct (ammonium sulfate- 50%) 10 ml liter^{-1} was used. All other fertilizers (N, P, and K) were applied according to the ND fertilizer recommendation tables and equations (Franzen, 2018). Mono-ammonium phosphate (MAP) and potassium chloride were used to supply P and K, respectively. Recommended N rate of 180 kg ha^{-1} was applied using urea as a fertilizer source and total N application rate was balanced considering the N supply from residual soil N (0-15 and 15-60 cm depths) and the N contained in different S application rates. All fertilizers were applied immediately prior to planting and incorporated 3 inches deep using a field cultivator operated at 10 kmh.

2.3.3. Tissue sampling

Eight random samples were taken from the middle four rows of each experimental unit at V6 and V12 growth stages during the 2016 growing season. Whole corn plants were collected at the V6 stage and first fully mature leaves were collected at V12. In 2017, the first plant sampling was conducted at the V6 stage and the second sampling was conducted at the early reproductive state (R1). At R1, eight ear leaves were collected from the middle four rows. Tissue samples were dried at 60ºC, ground to pass through a 2-mm sieve, and analyzed for S concentration using inductively couple plasma emission spectroscopy (ICP) (Thermo Scientific-ICAP 6500, Thermo Fisher Scientific, Waltham, MA, USA). Total N in these samples was determined using an automated CNS combustion analyzer (Elementar America Inc., Ronkonkoma, NY, USA).

2.3.4. Yield analysis

Corn grain yield was obtained by hand harvesting the middle two rows from each experimental unit within the producer field sites, and middle three rows in the case of research fields using a plot combine (ALMACO). Grain moisture and test weight were measured using Dickey-John Grain Moisture tester (GAC 500 XT). Final grain yield was adjusted to 155 g kg^{-1} moisture content before recording and data analysis.

2.3.5. Statistical analysis

Statistical analyses were conducted using SAS 9.4 and SAS enterprise guide 6.1 (SAS Institute, 2013, Cary, NC, USA). Analysis of variance (ANOVA) was calculated by SAS PROC GLM procedure to determine the significance of S treatments and its interaction with location. Means of S treatments were compared using Fisher's least significant difference (LSD) at the 95% confidence level. Linear regression analysis was conducted with PROC REG in SAS 9.4 and significant correlation coefficients (R^2) were reported.

2.4. Results and discussion

2.4.1. Location characteristics

Initial soil properties are presented in Table 1. Soil P availability varied across all locations; site 2 and 10 had low soil P (< 8 mg kg^{-1}), site 3, 5, and 6 had a medium range between 8 and 11 mg kg^{-1}, and the remaining five sites had values greater than 15 mg kg^{-1}. Soil K availability was low for site 2 and 6 (< 80 mg kg^{-1}), medium for site 9 (80-150 mg kg^{-1}), with higher values for the remaining seven sites (> 150 mg kg^{-1}). Five sites (2, 3, 6, 7, and 9) tested medium in soil organic matter (SOM) (30-40 g kg^{-1}) and the other five sites had higher SOM (40 g kg^{-1}). Initial extractable SO_4-S at 0-15 cm soil depth ranged from 7 to 19 mg kg^{-1}. A range of soil textures was considered during the site selection as S deficiency can be found on both light textured (Fox et al., 1964; Reneau, 1983; Rehm, 2005) and higher clay textured soils (O'Leary and Rehm, 1990). Three soils (4, 5, and 8) were fine textured; other six (1, 2, 6, 7, 9, and 10) were medium textured and one site (3) had a coarse textured soil.

Table 1. Geographical locations and initial soil properties of fields used to determine corn response to incremental S application rates during 2016-17 growing seasons.

Sites		Latitude and Longitude	Soil series	Texture	Previous crop	NO_3-N[†] (kg ha^{-1})	P (mg kg^{-1})	K (mg kg^{-1})	pH (1:1)	EC (mmhos cm^{-1})	OM (g kg^{-1})	SO_4-S (mg kg^{-1})
1	Absaraka	46°58'42.6" N 97°25'20.9" W	Glyndon	Silt loam	Soybean	112	25	166	8.1	0.69	49	16
2	Ada I	47°18'53.8" N 96°24'31.8" W	Wheatville	Loam	Wheat	26	3	68	8.2	1.21	31	18
3	Downer I	46°48'06.2" N 96°32'52.1" W	Elmville	Sandy loam	Soybean	64	11	194	8.6	2.36	38	19
4	Gardner	47°09'55.3" N 97°03'14.9" W	Fargo	Silty clay loam	Soybean	11	15	186	6.9	1.37	46	11
5	Walcott I	46°31'45.2" N 96°54'14.3" W	Fargo	Silty clay	Soybean	78	11	378	7.7	0.54	42	14
6	Ada II	47°21'20.5" N 96°25'43.0" W	Augsburg	Loam	Soybean	59	9	74	7.0	1.15	31	16
7	Amenia	46°59'05.5" N 97°14'26.4" W	Glyndon-Tiffany	Silt loam	Soybean	83	22	246	7.3	0.80	36	10
8	Casselton	46°56'53.8" N 97°12'10.5" W	Bearden	Silty clay loam	Soybean	91	20	217	7.4	0.46	46	7
9	Downer II	46°51'55.8" N 96°30'55.0" W	Lamoure	Silt loam	Soybean	82	13	96	7.2	0.60	33	15
10	Walcott II	46°31'05.5" N 96°52'24.1" W	Wheatville	Silt loam	Soybean	45	7	188	8.0	0.48	46	13

[†] NO_3-N up to 60-cm, all other properties were determined up to 15-cm

Total precipitation during the 2016 and 2017 growing season is shown in Figure 2. The cumulative precipitation during May through September was higher in 2016 compared to 2017 at all sites. Except for site 2 in 2016, all other sites received less rainfall than the normal rainfall. Site 2 got 13 cm more rainfall whereas the departure from the normal precipitation was 1.6 cm, 9.7 cm, 1.6 cm, and 9.7 cm for sites 1, 3, 4, and 5, respectively. All sites showed a dry period in May and June during 2016 growing season while most of the precipitation occurred in July. In 2017, the cumulative precipitation for all sites was much lower than the normal mean annual precipitation. The actual precipitation was 7.1 cm, 14.3 cm, 14.3 cm, 15.3 cm, and 17.8 cm less than the normal precipitation for sites 6, 7, 8, 9, and 10, respectively. A dry period occurred in May (all sites), July (all sites except 6) and August (sites 6, 7, and 8) in 2017.

The air temperature and growing degree-days across all sites during 2016 and 2017 growing season is presented in Table 2 and Table 3, respectively. Soils were warmer in 2016 growing season compared to 2017 growing season. In 2016, the average air temperature (21ºC) from May through September was more than the normal mean air temperature (18.3ºC) for all sites. It was 1.6ºC, 1.1ºC, 4.2ºC, 3.9ºC 0.4ºC more than the normal temperature for sites 1, 2, 3, 4, and 5 in during 2016. In 2017, the mean air temperature (18.6ºC) for all sites was near normal (18.3ºC) for all sites.

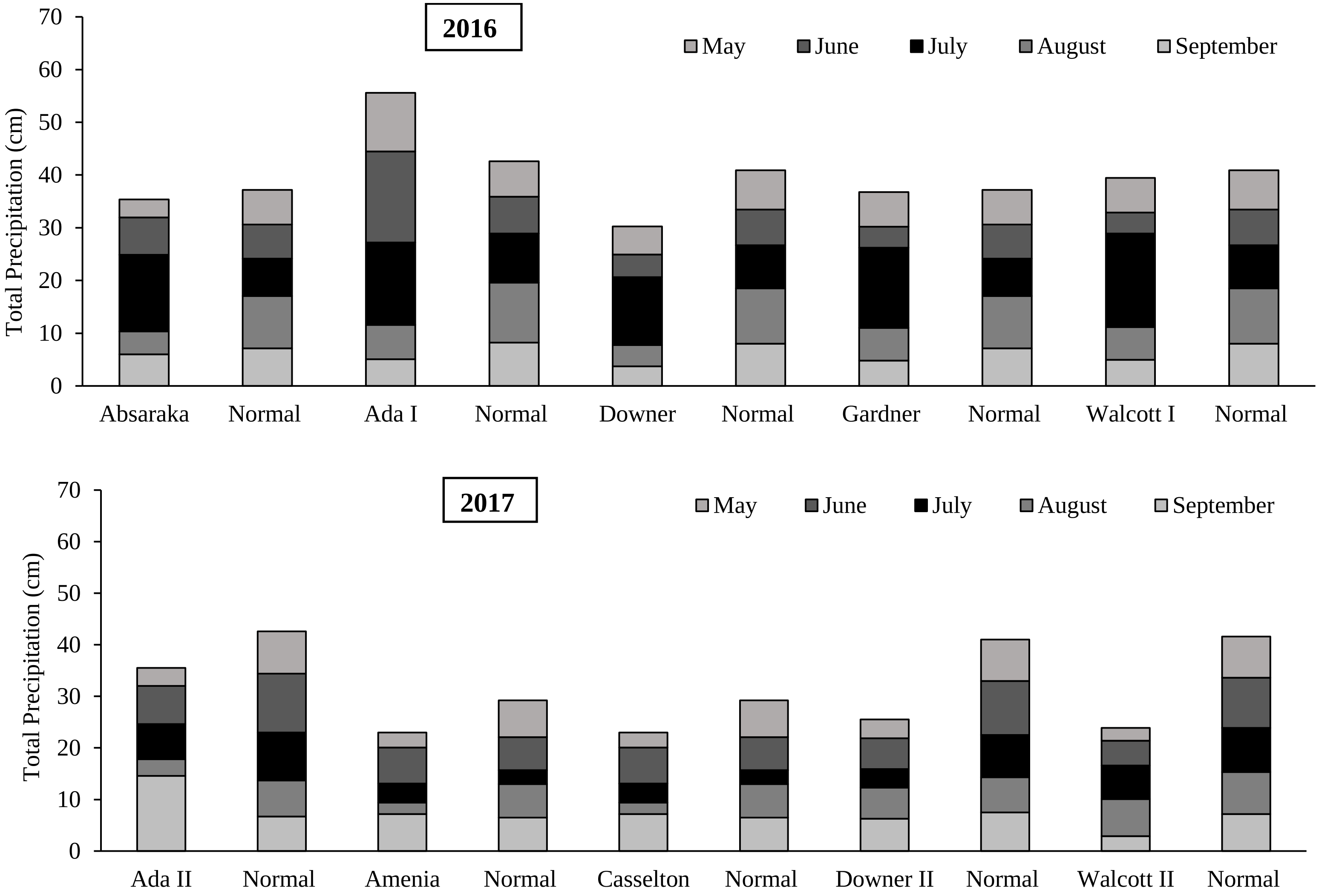


Fig. 2. Monthly precipitation (cm) for ten experimental sites during 2016 and 2017 growing seasons.

Table 2. Actual air temperature and normal air temperature (1991-2016) for ten experimental sites during 2016 and 2017 growing seasons.

Year	2016									
Sites	1		2		3		4		5	
	Absaraka		Ada I		Downer I		Gardner		Walcott I	
Air temperature	Actual	Normal	Actual	Normal	Actual	Normal	Actual	Normal	Actual	Normal
Months	---------------°C---------------									
May	16.2	14.0	14.6	13.4	23.4	14.6	16.2	14.0	15.1	14.6
June	20.7	19.0	19.0	18.5	27.1	19.6	20.7	19.0	20.2	19.6
July	22.4	21.8	21.3	21.3	28.2	22.4	22.4	21.8	21.8	22.4
August	21.3	20.7	20.2	20.2	27.8	21.3	21.3	20.7	20.7	21.3
September	17.4	15.1	15.7	14.6	23.0	15.7	17.4	15.1	16.8	15.7
	2017									
	6		7		8		9		10	
	Ada II		Amenia		Casselton		Downer		Walcott II	
	Actual	Normal	Actual	Normal	Actual	Normal	Actual	Normal	Actual	Normal
Months	---------------°C---------------									
May	13.4	13.4	14.0	14.0	14.0	14.0	15.7	14.6	15.1	14.6
June	19.0	19.0	21.3	19.0	21.3	19.0	20.2	19.6	19.6	19.6
July	21.3	21.3	23.5	21.8	23.5	21.8	21.8	22.4	22.4	22.4
August	18.5	20.2	19.0	20.7	19.0	20.7	20.7	21.3	21.3	21.3
September	15.7	14.6	15.1	15.1	15.1	15.1	16.8	15.7	16.2	15.7

Table 3. Growing Degree Days (GDD) for ten experimental sites during 2016-17 growing season.

Year	2016					2017				
Sites	1	2	3	4	5	6	7	8	9	10
	Absaraka	Ada I	Downer I	Gardner	Walcott I	Ada II	Amenia	Casselton	Downer II	Walcott II
Days after sowing	---GDD--									
15	282	260	255	275	290	113	142	142	142	149
29	670	668	673	665	672	172	213	213	208	219
43	935	915	902	940	925	428	499	499	488	502
57	1228	1350	1412	1250	1370	605	702	702	687	700
78	1628	1750	1735	1709	1701	972	1126	1126	1080	1118
92	2155	2159	2164	2165	2153	1271	1452	1452	1389	1447
106	2347	2327	2332	2305	2345	1479	1688	1688	1613	1683
123	2835	2778	2787	2745	2733	1736	1972	1972	1873	1965
138	2915	2927	2845	2825	2813	1935	2207	2207	2091	2200

2.4.2. Corn grain yield

Corn yield response to different S application rates for ten sites during the 2016-17 growing season is presented in Table 4. During 2016, corn yield ranged between 10.2 Mg ha^{-1} (with check treatment at site 1) to 16.7 Mg ha^{-1} (with 33 kg S ha^{-1} at site 5). Significant increase in corn yield occurred at only one site (site 3), where application of 33 kg S ha^{-1} increased corn yield 3.4 Mg ha^{-1}. Response to S for a coarse textured soil at site 3 agrees closely with the results from previous studies (Fox et al., 1964; Daigger and Fox, 1971, and Rehm, 1984). Corn response to S was not significant at other four sites. During the 2017 growing season, corn yield varied from 6.05 Mg ha^{-1} to 15.4 Mg ha^{-1} both under check at site 9 and 10, respectively. Compared to check plots, only one site (site 9) showed a significant yield increase of 1.3 Mg ha^{-1} with application of 44 kg S ha^{-1}. None of the other sites showed significant yield increase with S fertilizer application. At site 1 in 2016 and site 6 in 2017, corn showed S deficiency at early growth stages, but it disappeared over time as S become available from organic matter. Higher application of S (44 kg S ha^{-1}) resulted in a yield decrease at site 10 in 2017 and site 4 in 2016. This might be due to deficiency of other nutrients in the soil such as zinc that resulted in lower yield in treated plots.

Kaiser et al., (2010) found that usually soils with medium organic matter (20-40 g kg^{-1}) respond to S application. This was not observed in our study, as sites 2, 6, and 7 did not show response to S even though these were medium in soil organic matter. Further, no response occurred in high organic matter soils (40 g kg^{-1}). Considering the textural class of sites, grain yield increased at one coarse textured and one medium textured soils. This is in agreement with

Table 4. Corn grain yield (Mg ha^{-1}) response to incremental S application rates at ten sites during 2016-17 growing season.

Year	2016					2017				
Site	1	2	3	4	5	6	7	8	9	10
Treatment (S kg/ha)	Absaraka	Ada I	Downer I	Gardner	Walcott I	Ada II	Amenia	Casselton	Downer II	Walcott II
0	10.2 a[†]	14.9 a	11.5 a	12.7 b	15.0 a	12.4 ab	10.8 a	13.9 ab	6.05 a	15.4 b
11	11.4 a	15.5 a	12.7 ab	11.1 a	14.1 a	13.1 b	11.1 a	14.0 ab	6.89 b	14.9 ab
22	12.2 a	14.8 a	12.5 ab	11.2 a	13.5 a	12.1 a	10.8 a	13.6 a	6.69 ab	15.2 ab
33	11.1 a	15.4 a	14.9 b	11.7 ab	16.7 a	12.4 ab	10.9 a	14.0 ab	6.88 b	14.4 ab
44	12.4 a	14.7 a	13.4 ab	10.9 a	14.2 a	12.0 a	10.4 a	14.5 b	7.32 b	13.8 a

ANOVA

Source of Variation	P value
Site	<0.0001
Year	<0.0001
Treatment	0.49 ^{ns}
Site*treatment	0.71 ^{ns}
Year*treatment	0.43 ^{ns}
Treatment*site*year	<0.0001

[†] Means followed by the same letter within a column are not significantly different at $P < 0.05$ according to Fisher's least significant difference (LSD) test.

ns means non-significant at $P < 0.05$

other works that reported responses to S in medium textured soils (Rehm, 2005; O'Leary and Rehm, 1990).

Overall, only two out of ten sites responded to applied S fertilizer. During both years, the initial growth months (May-June) received very low rainfall, which led to less S leaching from the soils. Since the ND sub-soils are rich in sulfate salts, this dry season could have resulted in S accumulation in surface layer with upward movement of water and its solutes. The S from sub-soil combined with mineralized S from organic matter may have fulfilled the crop S demand. Some S may be available from herbicide mixture since it has 50% ammonium sulfate salt. More sites would have responded to S if the initial growing season had higher rainfall. In addition, research in Minnesota for corn indicated a greater advantage to band application of S (Kim et al., 2013) when considering the yield and S removal. Thus, the method of fertilizer application might have an impact on the crop response.

2.4.3. Plant S concentration

Plant S concentrations with S application for 2016-17 growing season are presented in Table 5. During 2016, the tissue S concentration varied from 2.2 g kg^{-1} to 2.7 g kg^{-1} and from 1.9 g kg^{-1} to 2.5 g kg^{-1} in V6 and V12 growth stages, respectively. No significant increase in tissue S concentration from fertilizer S was observed at V6 and V12 growth stage. Site 3 resulted in highest tissue S in checks as compared to the treated plots. In 2017, tissue S concentration varied from 1.9 g kg^{-1} to 2.8 g kg^{-1} in V6 stage and from 1.6 g kg^{-1} to 3.3 g kg^{-1} at the R1 stage. Two (site 6 and 10) of five sites showed significant increase in tissue S concentration during the V6 stage, with an increase of 0.6 g kg^{-1} with S fertilizer application. Sulfur application did not increase ear leaf S concentration at any site, which has previously been reported by Hoeft et al., (1985), O'Leary and Rehm, (1990), Stecker et al., (1995), and Sutradhar et al., (2017).

Table 5. Total tissue S (g kg^{-1}) with incremental S application rates at V6 and V12 growth stages in 2016 and at V6 and R1 growth stages of corn in 2017 growing season.

Year	2016									
Sites	1		2		3		4		5	
	Absaraka		Ada I		Downer I		Gardner		Walcott I	
Crop growth stage	V6	V12	V6	V12	V6	V12	V6	V12	V6	V12
Treatment (S kg ha^{-1})										
0	2.4 a[†]	2.4 a	2.6 a	2.0 a	2.6 a	2.2 b	2.4 a	2.1 ab	2.3 a	2.0 a
11	2.3 a	2.4 a	2.5 a	2.0 a	2.5 a	2.1 ab	2.3 a	2.5 b	2.4 a	1.9 a
22	2.5 a	2.1 a	2.4 a	2.1 a	2.5 a	2.1 ab	2.3 a	2.0 a	2.4 a	2.0 a
33	2.5 a	2.5 a	2.5 a	2.1 a	2.5 a	2.0 a	2.2 a	1.9 a	2.4 a	2.0 a
44	2.4 a	2.3 a	2.4 a	1.9 a	2.7 a	2.1 ab	2.5 a	1.9 a	2.5 a	1.9 a
	2017									
	6		7		8		9		10	
	Ada II		Amenia		Casselton		Downer		Walcott II	
	V6	R1	V6	R1	V6	R1	V6	R1	V6	R1
0	2.2 a	1.7 a	2.7 a	2.0 a	2.4 a	2.1 a	2.6 a	2.0 a	1.9 a	3.3 a
11	2.5 ab	1.8 a	2.4 a	1.8 a	2.7 a	1.9 a	2.8 a	2.1 a	2.4 ab	2.9 a
22	2.5 ab	1.7 a	2.6 a	1.9 a	2.3 a	1.6 a	2.8 a	1.9 a	2.3 ab	2.7 a
33	2.8 b	1.8 a	2.1 a	1.9 a	2.5 a	1.7 a	2.5 a	2.0 a	2.2 ab	3.1 a
44	2.5 ab	1.7 a	2.6 a	1.7 a	2.8 a	2.1 a	2.4 a	2.0 a	2.5 b	2.7 a

[†] Means followed by the same letter within a column are not significantly different at $P < 0.05$ according to Fisher's least significant difference (LSD) test.

Table 6. Corn plant S uptake (g plant^{-1}) at V6 stage for the 2017 growing season.

	2017				
	6	7	8	9	10
Sites	Ada II	Amenia	Casselton	Downer II	Walcott II
Treatments (S kg/ha)	S uptake (g plant^{-1})				
0	3.31 a[†]	2.65 a	1.77 a	3.29 a	2.67 a
11	4.05 a	2.59 a	2.36 a	2.77 a	2.81 a
22	3.45 a	2.71 a	1.59 a	3.39 a	2.88 a
33	4.84 a	2.16 a	1.88 a	2.43 a	2.86 a
44	4.27 a	2.88 a	1.76 a	2.14 a	2.93 a

†Means followed by the same letter within a column are not significantly different at $P < 0.05$ according to Fisher's least

In addition, there was no significant difference in S uptake at V6 crop stage in 2017 growing season (Table 6). Lockman, (1969) considered 2.0 g kg^{-1} S sufficient for small whole corn samples. In our study, the tissue S concentrations at V6 stage were greater than 2.0 g kg^{-1} except for the check treatment at site 10. Several critical S concentration values for R1 stage have been reported in the literature. Reneau, (1983) and Bryson et al., (2014) reported critical ear leaf S concentration was in the range between 1.5-1.7 g kg^{-1}; whereas Sawyer et al., (2011) suggested a wide sufficiency range of 1.0 2.1 g kg^{-1} at silking. All our R1 leaf S values were within this range except for site 10, which showed much greater values. In addition, V6 S concentration values for both years were greater than V12 and R1 values (except for site 10) in 2016 and 2017, respectively. Decline in tissue concentration with greater maturity was expected due to the dilution effect (Robson et al., 1995).

2.4.4. Relationship between corn yield and soil properties

Sulfate S extracted from the soil has been used effectively for predicting S need for corn production (Fox et al., 1964; Kang and Osiname, 1976, and Stecker et al., 1995). However, poor relationships between soil sulfate and crop performances are commonly reported (Scherer, 2009; Sawyer, 2011 and Franzen, 2015). In our experiment, soil S within 0-30 cm varied from 17.06 to

60.17 mg kg^{-1} at V6 stage and from 10.35 to 52.88 mg kg^{-1} at R2 stage in the 2016 growing season. There was no relationship between soil sulfate and corn yield. In a study in Minnesota, Kim et al., (2013) reported a negative relationship between soil S and corn yield since lower yield was noticed in the soil with highest S concentration. Further, Stecker et al., (1995) observed better correlation coefficient when 0-30 cm soil S was considered instead of top 0-15 cm soil S. However, we did not see such results in our current study. The poor correlation of corn yield and soil S in our study supports the observations of O'Leary and Rehm, (1990) and Hoeft et al., (1985), that soil S is not a reliable predictor in soils.

In contrast to soil S, soil organic matter has been considered a better tool in predicting corn yield (Zhao et al., 1999). Generally, corn grown on low organic matter soils respond to S. In the current study, five soils had SOM in the medium range and other five had high SOM. A positive correlation between corn yield and SOM has been reported by Kim et al., (2013), indicating a high yield in high OM soils. In contrast, no clear relationship was noticed between SOM and corn yield in our experiment (data not shown). Similarly, recent work by Sutradhar et al., (2017) reported a non-significant relationship among these variables. In addition, a study in Iowa did not show an impact of SOM on responsive and non-responsive sites (Sawyer et al., 2011). This uncertainty can be due to some other factors like environment, crop and soil type.

2.4.5. Relationship between corn yield and tissue S

Relationship between tissue S and corn yield and N/S and corn yield are presented in Figure 3 and 4, respectively. A significant relationship was observed between tissue S concentration and corn yield in V6 and V12 crop stage during 2016, and V6 and R1 stage during 2017. Among all, R1 tissue S and corn yield showed better correlation. In a recent study, Sutradhar et al., (2017) reported a strong relationship between corn yield and ear leaf S concentration. In contrast, Sawyer et al., (2011) found no clear relationship between ear leaf S and yield.

For corn, Stewart and Porter, (1969), Terman et al., (1973), and Reneau, (1983), have reported the successful use of N/S ratios in S deficiency diagnosis. Jones et al., (1980) considered N/S more stable through the growing season than is tissue S. The N/S ratio of 16:1 is said to be critical for corn crop (Stewart and Porter, 1969; Mortvedt, 1981; Reneau, 1983). In this experiment, the N/S ranged from 12:1 to 17:1 in 2016 and from 13:1 to 20:1 in 2017 growing season. Weil and Mughogho, (2000), have reported significant correlation between corn yield and N/S. In contrast, non-significant correlation was found between these parameters in the current study. Similarly, Daigger and Fox, (1971), Kang and Osiname, (1976), and Rehm, (1984) found that N/S ratios did not adequately predict the response of corn to S fertilizer.

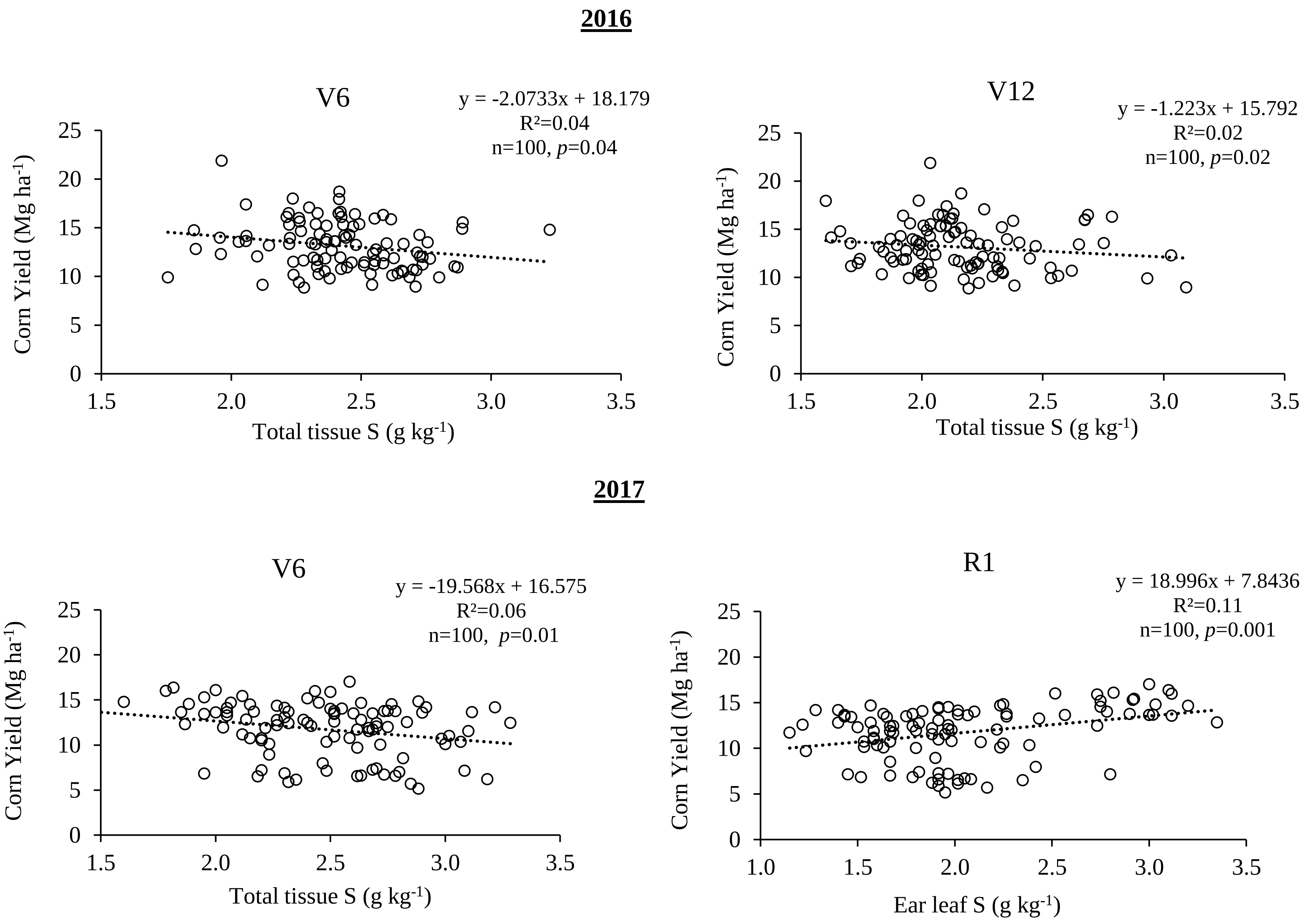
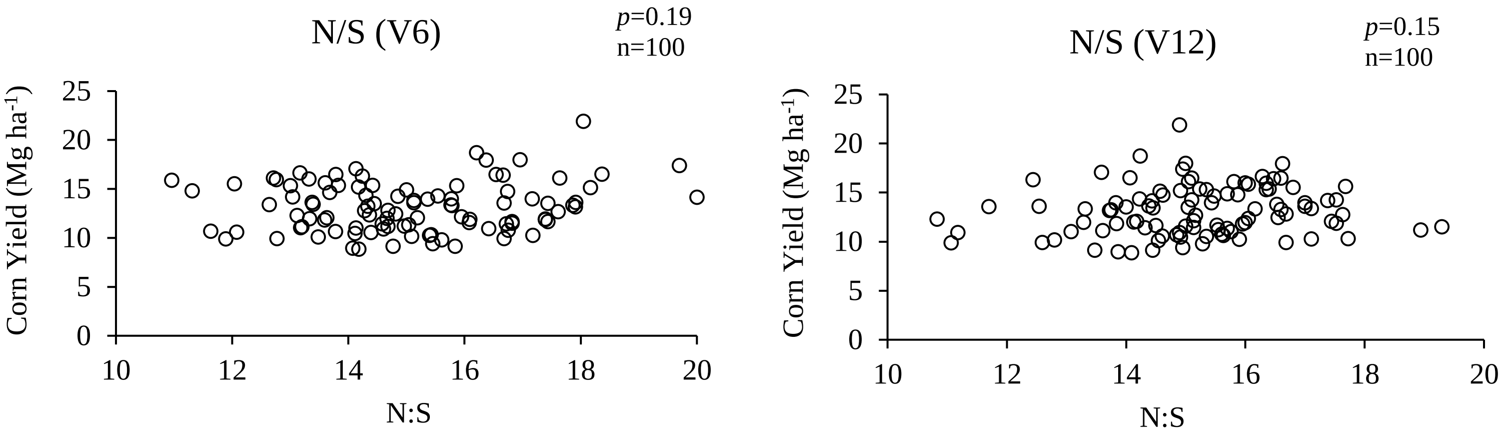


Fig. 3. Relationship between total tissue S (g kg^{-1}) and corn yield (Mg ha^{-1}) for 2016 and 2017 growing seasons.

2016



2017

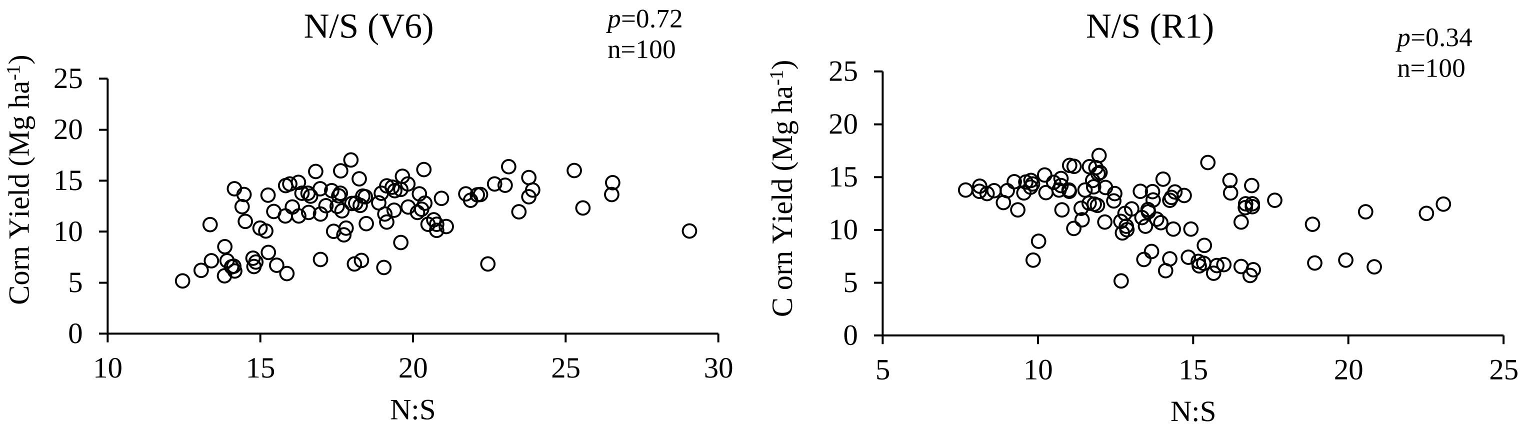


Fig. 4. Relationship between tissue N/S ratio and corn yield (Mg ha^{-1}) for 2016 and 2017 growing seasons.

2.5. Summary

Corn responded to S fertilizer at 2 of 10 sites. The S from other sources (organic matter, crop residues and sub-soil) might was sufficient for the other 8 sites. Further, the weather conditions played the significant role in determining the response. The results might have been difference if the seasons had received greater early-season rainfall. Based on these results, the decision to not consider soil sulfate soil tests as a diagnostic tool is justified. The soil and plant indicators did not accurately predict corn response. Although tissue S was significantly related to yield, the correlation value was very low leading to doubts regarding the reliability of the relationship. This study did not look at groundwater and sub-surface S that might be important parameters for future considerations. Overall, we concluded that corn response to S varies from soil to soil and no single parameter could estimate S deficiency and response to fertilizer S.

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3. ESTIMATION OF NITROGEN AND SULFUR MINERALIZATION IN SOILS AMENDED WITH CROP RESIDUES

3.1. Abstract

Predicting N and S mineralization of crop residues from the preceding crop might be a useful tool for forecasting soil N and S availability. This 8-wk incubation study was conducted with an objective to estimate N and S mineralization from crop residues. Two soils from eastern North Dakota and residues from three crops - corn (*Zea mays* L.), spring wheat (*Triticum aestivum* L.), and soybean (*Glycine max* L.) were used. The cumulative N and S mineralized from crop residues were fit to a first order kinetic model. Cumulative N mineralized ranged between 0.34 to 2.15 mg kg^{-1} and from 0.45 to 3.41 mg kg^{-1} for the Glyndon and Fargo soils, respectively. The un-amended soil showed highest N mineralization than the residue treatment for both soils. For S, highest mineralization occurred in the un-amended Glyndon soil (38.5 mg kg^{-1}) and in spring wheat-amended soil (5.30 mg kg^{-1}) at Fargo. There was no clear relationship between residue properties and mineralized N and S. This incubation study indicate that crop residue additions early in the growing season can have a negative impact on plant available nutrients due to immobilization of N and S during the time when crops need the nutrients most.

3.2. Introduction

In recent years, the Northern Great Plains have experienced a shift from traditional small grains (spring wheat (*Triticum aestivum* L.), durum wheat (*Triticum durum* Desf.) and barley (*Hordeum vulgare* L.)) and sunflower (*Heliantus annus* L.) production to corn (*Zea mays* L.) and soybean (*Glycine max* L.) production. In addition to a changing climate and economics that favor such a shift, high yielding, short season (< 100-day maturity) corn varieties are preferred compared to other traditional crops. The ease of weed control until lately, and the lack of susceptibility to disease compared to more traditional crops has also contributed to the shift by farmers in their crop choices. Adapted crop varieties along with reduced tillage systems combined with cold winters and cool, moist springtime pre-planting conditions have raised concerns about the availability of nutrients such as N and S becoming available from preceding crop residues in time to benefit the current crop. In addition, S deficiencies are being diagnosed in soils where S deficiencies previously had not been observed prior to production of high yielding crops.

Crop residues in agricultural soils provide the energy, carbon, and nutrients for microbial growth and activity, acts as a driving force for the mineralization-immobilization process in the soil and is a source of N for plants (Jansson and Persson 1982). Nitrogen mineralization is a vital component of the soil N cycle (Shukla et al. 2000) in which organic N forms are converted into plant-available, inorganic N forms. In addition to N, mineralization of crop residues can release inorganic S for subsequent crop uptake.

Sulfur is an important plant nutrient responsible for the synthesis of proteins and several vitamins and cofactors in plants (Kertesz and Mirleau 2004; Churka Blum et al. 2013). Increasing S deficiency in the world is becoming a major constraint for the crop production (Eriksen et al. 2004; Girma et al. 2005; Franzen 2015). The main reasons for increasing S deficiencies are the reduction of atmospheric S gas emissions in industrial areas, the increasing use of low S fertilizers, and increased S removal by high-yielding crop varieties (Scherer 2001; Franzen 2015). Since about 95% of total soil S accumulated from manures, crop residues, and fertilizers in soils is in organic forms (Ghani et al. 1991; Nguyen and Goh 1992), the mineralization of organic S from soil organic matter and crop residues becomes an increasingly important S source for plant uptake.

The mineralization of crop residues returned to soils is controlled by numerous factors including soil temperature, water, and biochemical composition of crop residues (Abiven et al. 2005; Khalil et al. 2005) including total N, C/N ratio, lignin content, as well as polyphenol content and their interaction (Nakhone and Tabatabai 2008; Vahdat et al. 2011; Abera et al. 2012). In the case of S, initial S content of residues (Janzen and Kucey 1988) and residue C/S ratios are important indicators of mineralization.

Stanford and Smith (1972) proposed long-term incubation studies to estimate potentially mineralizable N. The S mineralization is associated closely with N mineralization, as both nutrients are present in the organic pool of most soils within the same compounds (Zhou et al. 1999; Kellogg et al. 2006). Therefore, the methods to estimate N mineralization potentially can be used to estimate S mineralization (Niknahad Gharmakher et al. 2009). The results of these experiments might lead to better predictability of future plant available N and S from crop residues, which might aid in fertilizer management.

This study evaluated the potential mineralization of N and S from crop residues for two contrasting, highly productive soils used in corn production in eastern North Dakota. The main objectives of our study were: 1) to quantify the amount of N and S mineralized or immobilized from different crop residues commonly found in most eastern North Dakota cropping systems; 2) to estimate potentially mineralizable N and S during the early to mid-growth cycle of most eastern North Dakota crops; and, 3) to evaluate the relationship between mineralized N and S and residue characteristics.

3.3. Materials and methods

3.3.1. Study area

The study area was located in the Red River Valley of the North in eastern North Dakota in Major Land Resource Area 56 (USDA- NRCS 2006) as part of ongoing research to evaluate corn response to S fertilizer. The soils in this area are of lacustrine origin in a nearly level lake plain. Average annual precipitation is 475 to 550 mm with more than 50% of the annual precipitation occurring during the growing season (April-September). The average annual temperature is 2 to 7 ºC and decreases from south to north. The average frost-free period is 105 to 135 days. Corn and soybean have become the dominant crops in this region during the last decade although small grains (spring wheat and barley) are still frequently grown within predominantly corn-soybean cropping systems.

3.3.2. Soils

Composite bulk soil samples to a 15-cm depth were collected from agricultural fields near Absaraka (46°58'42.6"N, 97°25'20.9"W) (Glyndon soil series) and Walcott (46°31'45.3" N, 96°54'14.3"W) (Fargo soil series) of North Dakota in fall 2016. The Glyndon soils were coarse-silty, mixed, frigid Aeric Calciaquolls (sand: 20.1%, silt: 55.4%, and clay: 24.5%) and Fargo soils were fine, smectitic, frigid Typic Epiaquerts (sand: 9.10%, silt: 42.8%, and clay: 48.1%). Both sites have been under corn and soybean production, and were chosen based on the difference in texture, pH and organic matter and were adjacent to fields utilized in other fertility studies. Soil physical and chemical characteristics are reported in Table 1.

The bulk soils were air-dried, mechanically crushed and passed through a 2-mm sieve before analysis for basic physio-chemical properties. Soil texture was determined using a hydrometer method (Gee and Bauder 1986), and pH was determined using 1:2 (w:v) soil to water ratio (Watson and Brown 1998). Total soil organic matter was measured using the weight loss on ignition method (Combs and Nathan 1998). Soil samples were extracted with water and analyzed for NO_3-N (Gelderman and Beegle 1998). Total N, total C, and total S were determined by high temperature combustion using an Elementar CNS Elemental Analyzer (vario MACRO cube) (Elementar 2007). For sulfate S, soil samples were extracted with calcium monophosphate and analyzed using a turbidimetric method (Combs et al. 1998).

3.3.3. Crop residues

Three crop residues were selected for this study (corn (*Zea mays* L.), spring wheat (*Triticum aestivum* L.), and soybean (*Glycine max* L.)) because these are the major crops grown in the study region. Random plant samples (whole plants excluding roots) across the field were collected in three replicates during the crop harvest. The residues were oven-dried at 60°C until dry and ground to 1-mm size in a Wiley mill. One-half of the residues were used for incubation study and the other half was kept for estimation of total carbon, total N, and total S. The C and N analyses were done by high temperature combustion analysis on the Elementar CNS Elemental Analyzer described above (Elementar, 2007). For S content, samples were digested with nitric acid and hydrogen peroxide in a CEM Mars microwave system and then analyzed by inductively couple plasma emission spectroscopy (ICP) (Thermo Scientific-ICAP 6500).

3.3.4. Incubation experiment

3.3.4.1. Nitrogen mineralization

Laboratory incubations were carried out to determine N mineralization according to the procedure described by Stanford and Smith (1972). Fifteen grams of soil from each site was mixed with 15g of acid washed quartz sand and 0.5g of each crop residue in three replicates. The mixed samples were packed in 50 mL leaching tubes with a thin pad of glass wool below and above the soil mixture to prevent soil loss during leaching and soil disturbance during addition of the leaching solution. Prior to the incubation, the soil-sand mixture was leached to remove the initial mineral nutrients. For N leaching, 30 mL 0.01 mol $CaCl_2$ and 5 mL nutrient solution were used. The nutrient solution was composed of 0.002 mol $CaSO_4.2H_2O$; 0.002 mol $MgSO_4$; 0.005 mol $Ca(H_2PO_4)_2.H_2O$; and 0.0025 mol K_2SO_4 and did not contain N. The tubes were then incubated at 25°C for eight weeks. Periodic leaching was carried out with 30 mL 0.01 mol $CaCl_2$ followed by 5 mL nutrient solution at 0, 3, 5, 7, 14, 21, 28, 35, 42 and 56 days of incubation. Leachates were analyzed for total N ($NH_4^+ + NO_3^-$) using an ammonia auto-analyzer (TL-2800, Timberline Instruments, Boulder, CO, USA).

3.3.4.2. Sulfur mineralization

The incubation procedure for S mineralization was similar to N. Instead of three crop residues, only two residues (corn and spring wheat) were used for S. For initial and periodic S leaching, 30 mL distilled water was used, and no nutrient solution was added. The S in the leachates was analyzed by ICP.

3.3.5. Kinetics model

The first order kinetic equation given by Stanford and Smith (1972) was used for estimating the potentially mineralizable N (N_0) and S (S_0) as well as rate constants (k_1 and k_2).

$$N_{min} = N_0 (1-e^{-k_1 t}) \text{ and } S_{min} = S_0 (1-e^{-k_2 t})$$

Here, N_{min} = ppm N mineralized (cumulative) during time t (days); N_0 = N mineralization potential (ppm); S_{min} = ppm S mineralized (cumulative) during time t (days); S_0 = S mineralization potential (ppm), k_1 and k_2 are rate constants for N and S mineralization, respectively.

3.3.6. Statistical analyses

Statistical software SAS 9.4 and SAS Enterprise Guide 6.1 were used for data analyses (SAS Institute Inc. 2013). The first order model was fit to the cumulative N and S mineralized with different crop residues by a nonlinear regression procedure using Marquadt's method in SAS 9.4. Pearson correlation coefficients were used to evaluate the relationship between residue properties (total N, total S, total C, C/N, C/S and N/S) and cumulative N and S mineralized at $p < 0.10$ and $p < 0.05$.

3.4. Results and discussion

3.4.1. Basic soil and residue properties

Initial soil and residue characteristics are presented in Table 7. The soil textures were silt loam and silty clay for the Glyndon and Fargo soils, respectively. The soil pH ranged from slightly alkaline to moderately alkaline. The soil organic matter varied from 4.2 % to 4.9 % for the Fargo and Glyndon soils, respectively. The initial N concentration of both soils was similar whereas S concentration varied from 0.32 to 0.49 g kg^{-1} for the Fargo and Glyndon soils, respectively. The Fargo soil showed higher C/N, C/S and N/S ratios than the Glyndon soil.

Total N in the crop residues varied from 4.9 to 10.5 g kg^{-1}, with the lowest N content in soybean and the highest in corn residues. Total C content of residues followed the order soybean> spring wheat> corn and ranged between 418 to 431 g kg^{-1}, whereas S content followed the order spring wheat> corn> soybean, varying from 0.40 to 1.30 g kg^{-1}. The C/S, C/N and N/S ratios of crop residues ranged from 323 to 1077, 40.0 to 88.0 and 6.8 to 12.7, respectively.

Table 7. Basic physical and chemical characteristics of soils and nutrient concentrations of the residues used for the 56-d incubation study.

Characteristics	Soil		
	Glyndon		Fargo
Texture class	Silt loam		Silty clay
Soil series	Glyndon silt loam		Fargo silty clay
Taxonomic Classification	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls		Fine, smectitic, frigid Typic Epiaquerts
Sand (g kg^{-1})	201		910
Silt (g kg^{-1})	554		428
Clay (g kg^{-1})	245		481
pH	8.10		7.70
Organic matter (g kg^{-1})	49.0		42.0
Total C (g kg^{-1})	22.5		24.0
Total N (g kg^{-1})	2.20		2.30
Nitrate-N (mg kg^{-1})	30.0		16.0
Total S (g kg^{-1})	0.49		0.32
Sulfate-S (mg kg^{-1})	16.0		14.0
C/N ratio	10.2		10.4
C/S ratio	45.9		74.7
N/S ratio	4.49		7.19
	Residues		
	Corn	Soybean	Spring wheat
Total C (g kg^{-1})	418	431	420
Total N (g kg^{-1})	10.5	4.90	8.80
Total S (g kg^{-1})	0.83	0.40	1.30
C/N ratio	40.0	88.0	48.0
C/S ratio	504	1077	323
N/S ratio	12.7	12.3	6.80
	Standard values		
	Corn	Soybean	Spring wheat
C/N ratio	50	20	80
C/S ratio	350	125	300

3.4.2. Nitrogen mineralization

The cumulative N mineralized in soils ranged from 0.34 to 2.15 mg kg^{-1} and from 0.45 to 3.41 mg kg^{-1} for the Glyndon and Fargo soils, respectively (Fig. 5 and Table 8). The un-amended soils showed highest cumulative N mineralization at both sites. Nearly 0.09 % of total soil N for the Glyndon soil and 0.15 % for the Fargo soil was mineralized during the 8-week incubation period. Among treatments, the N mineralization followed the trend: corn > spring wheat > soybean for the Glyndon soil and spring wheat > corn > soybean for the Fargo soil. Expressed as a percent of added N, the amount of cumulative N mineralized was -0.016 %, -0.037 %, and -0.019 % for corn, soybean and spring wheat, respectively in Glyndon soils. In Fargo soils, it was -0.028 %, -0.063 %, and -0.033 % for corn, soybean and spring wheat, respectively.

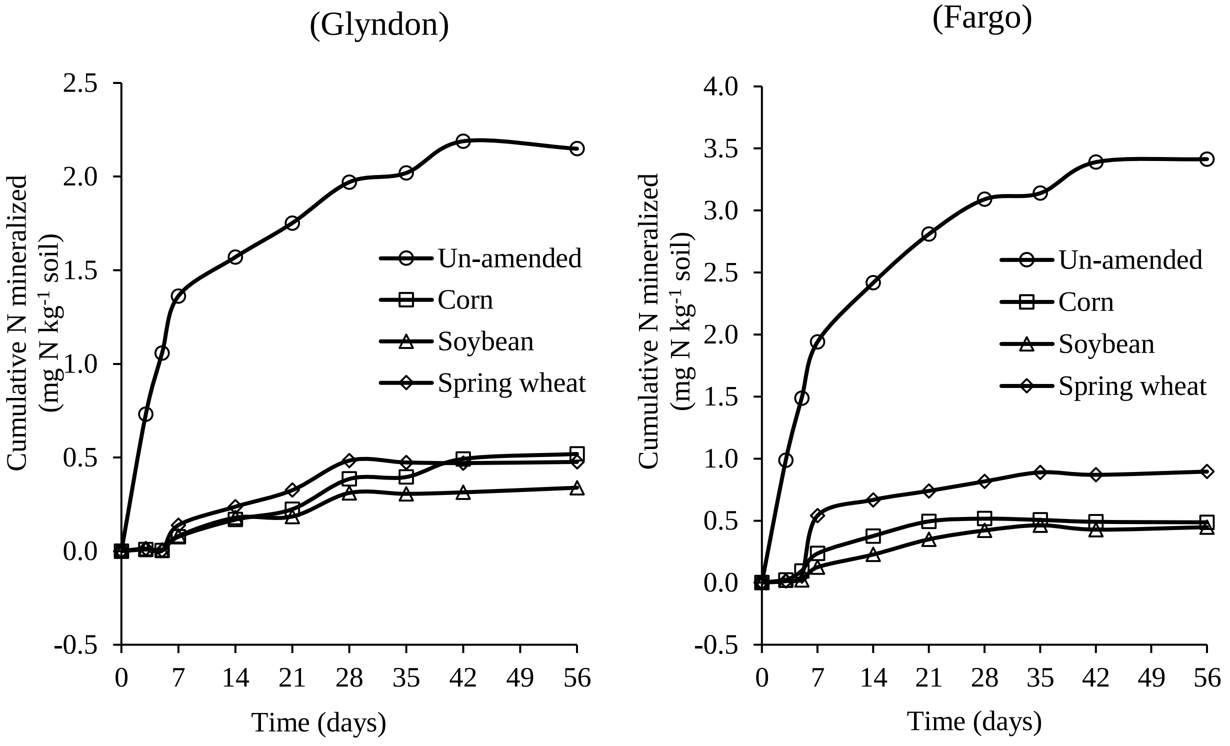

Fig. 5. Cumulative N mineralized in soils amended with crop residues after the 56-d incubation period for Glyndon and Fargo soils.

Table 8. Cumulative N mineralized (mg kg^{-1}), percent of added N mineralized, mineralization potential, and rate constant of soils amended with corn, soybean, and spring wheat residues after the 56-d incubation period for two contrasting soils.

Soil	Treatment	Cumulative mineralized N (mg kg^{-1})	Percent of added N mineralized[a]	Mineralization Potential ($N_{0)}$ (mg kg^{-1})	Rate Constant (k_1) (d^{-1})
Glyndon	Un-amended	2.15 (0.15)[b]	-	2.05 (0.11)	0.13
	Corn	0.52 (0.12)	-0.016[c]	1.32 (0.59)	0.01
	Soybean	0.34 (0.08)	-0.037	0.44 (0.11)	0.04
	Spring wheat	0.48 (0.18)	-0.019	0.63 (0.25)	0.05
Fargo	Un-amended	3.41 (0.93)	-	3.29 (0.76)	0.11
	Corn	0.49 (0.09)	-0.028	0.54 (0.07)	0.08
	Soybean	0.45 (0.04)	-0.063	0.55 (0.04)	0.04
	Spring wheat	0.89 (0.06)	-0.033	0.98 (0.11)	0.07

[a] $\frac{(\text{Total N mineralized in amended soil} - \text{total N mineralized in umamended soil}) \times 100}{\text{Amount of N added through residues}}$

[b] Parenthesis include standard deviation

[c] Negative values indicate net N immobilization.

In all treatments, the cumulative N mineralized was low during the first week, but it increased gradually afterward (Figure 5). Nourbakhsh, (2006) observed that there are two phases in inorganic N dynamics following corn residue application: the first two weeks in which net N immobilization occurs and rest of the incubation period in which net N mineralization happens. In our study, the N decline during the first week indicates net N immobilization followed by subsequent net N mineralization as the incubation proceeded.

The soils amended with crop residues had a lower cumulative N mineralization than the un-amended soils, which demonstrated that all the residue-amended soils immobilized N. Previously, Li et al., (2013) reported that cumulative mineralized N was 22-93% lower in residue treated soils as compared to un-amended soils on day 56. The C/N ratio and total N content of residues affect the N mineralization (Iritani and Arnold, 1960). Our observations confirmed the previously reported pattern that addition of residues with high C/N ratios and low N induce N immobilization during their decomposition in soils (Mendham et al., 2004; Muhammad et al., 2011). Consequently, in the current experiment, soybean residue addition resulted in greater N immobilization than other two residues. In the study by Abiven and Recous, (2007), the mineral N content in the soil was adjusted to 80 mg kg^{-1} to prevent N limitations for the decomposition process. In contrast, in our study, the absence of an N source other than residues most likely caused N limitation for the decomposer microorganisms.

The mineralization pattern of corn and spring wheat residues were quite different for the two soils. Even though the spring wheat residues had higher C/N ratio than corn, the cumulative mineralized N was low for corn residue for the Fargo soil. Previously, Broersma et al., (1999) found that N mineralization from barley was higher than that of fescue (*Festuca rubra* L.) even though barley (*Hordeum vulgare* L.) had a higher C/N ratio than fescue. The difference may be due to the types of C containing components in the crop residues. Lignin components will be more resistant for decomposition than carbohydrate and cellulose components. In addition, residue incorporation into soils can alter the microbial enzymatic synthesis and reactions (Deng and Tabatabai, 1996) and thus, this change in enzymatic activity altered the decomposition process and the related N cycling (Li et al., 2013).

3.4.3. Nitrogen mineralization constants

The N mineralization potential and their rate constants are presented in Table 8. The potentially mineralizable N (N_0) varied from 0.44 to 2.05 mg kg^{-1} for the Glyndon soil and from 0.54 to 3.29 mg kg^{-1} for the Fargo soil. At both sites, highest N_0 was found for un-amended soils, illustrating a stronger potential of these soils to mineralize N from organic matter. The rate constant for the Glyndon soil ranged from 0.01 to 0.13 day^{-1} and from 0.04 to 0.11 day^{-1} in the Fargo soil. For soils with added residue, our calculated k_1 values are in line with the findings of Kaboneka et al., (1997), who estimated that the rate constants of corn, soybean and spring wheat residues ranged from 0.04 to 0.12 per day and from 0.01 to 0.03 per day for rapid and slowly decomposable soil organic matter, respectively.

3.4.4. Relationship between N mineralization and residue properties

The cumulative mineralized N showed positive correlation with N/S ratio, C/S ratio, and total S in residues (Table 9). Previously, a positive correlation was reported between N mineralization and total N of plant residues (Vahdat et al., 2011). However, our study showed a non-significant correlation similar to that reported by Fox et al., (1990) as well as Palm and Sanchez, (1991). When only the Glyndon soil was considered, cumulative N mineralized was positively correlated with total N in the residues. Abbasi et al., (2015), have reported a negative correlation between cumulative mineralized N and C/N ratio. However, in our experiment such relationship was observed only for the Glyndon soils.

Table 9. Pearson correlation coefficients between cumulative N mineralized and crop residue properties for Glyndon and Fargo soils.

Site	Parameters	C/N	C/S	N/S	Total C	Total N	Total S
Glyndon	Cumulative N	***	ns	ns	ns	***	ns
Fargo	Cumulative N	ns	***	***	ns	ns	***

*** Significant at the 0.1 probability level.
† ns, non-significant.

3.4.5. Sulfur mineralization

Sulfur mineralization results are shown in Fig. 6 and Table 10. The total S mineralized varied from 29.6 to 38.5 mg kg^{-1} for the Glyndon and from 3.3 to 5.3 mg kg^{-1} for the Fargo soils, respectively. The S mineralization followed a trend: un-amended > corn > spring wheat for the Glyndon soils and spring wheat > corn = un-amended for the Fargo soils. Based on total soil S content, 7.85 % and 1.04 % of native soil S are mineralized in the un-amended Glyndon and Fargo soils, respectively. In the case of the amended Glyndon soil, corn mineralized -0.506 %

and spring wheat mineralized –0.685 % of total added residue S. In Fargo soils, the cumulative mineralized S for corn and un-amended soil was nearly the same whereas 0.152 % of added S was mineralized in case of spring wheat residues.

High rates of mineralization were observed during the first week. Except for spring wheat residues in the Fargo soil, all other residue treated soils showed less mineralization than the un-amended soils. The mineralization pattern of S is dependent on the type of organic residue available (Tabatabai and Chae, 1991). Sulfur is mineralized when the crop residue C/S ratio < 200 and is immobilized if ratio > 400 (Barrow, 1960). In our study, wide C/S ratio of corn and spring wheat residues resulted in S immobilization. Further, S mineralization was noticed for spring wheat residues with the Fargo soil that may be due to decomposition of crop residue at a rate at or greater than mineralization of native SOM. In one study, Islam and Dick, (1998) observed that incorporation of residues, even with a narrow C/S ratio rice straw residue (*Oryza sativa* L.), had significantly less SO_4 (plant available form) produced over the 12-week period than un-amended soil. Sulfur form present in the residues also influences its mineralization rather than residue initial S tissue concentration (Janzen and Kucey, 1988; Wu et al., 1993; Singh et al., 2006; Niknahad-Gharmakher et al., 2012) so differences in results may be due to different S forms (ester-S and C-bonded S) and their transformations during the decomposition of the residues.

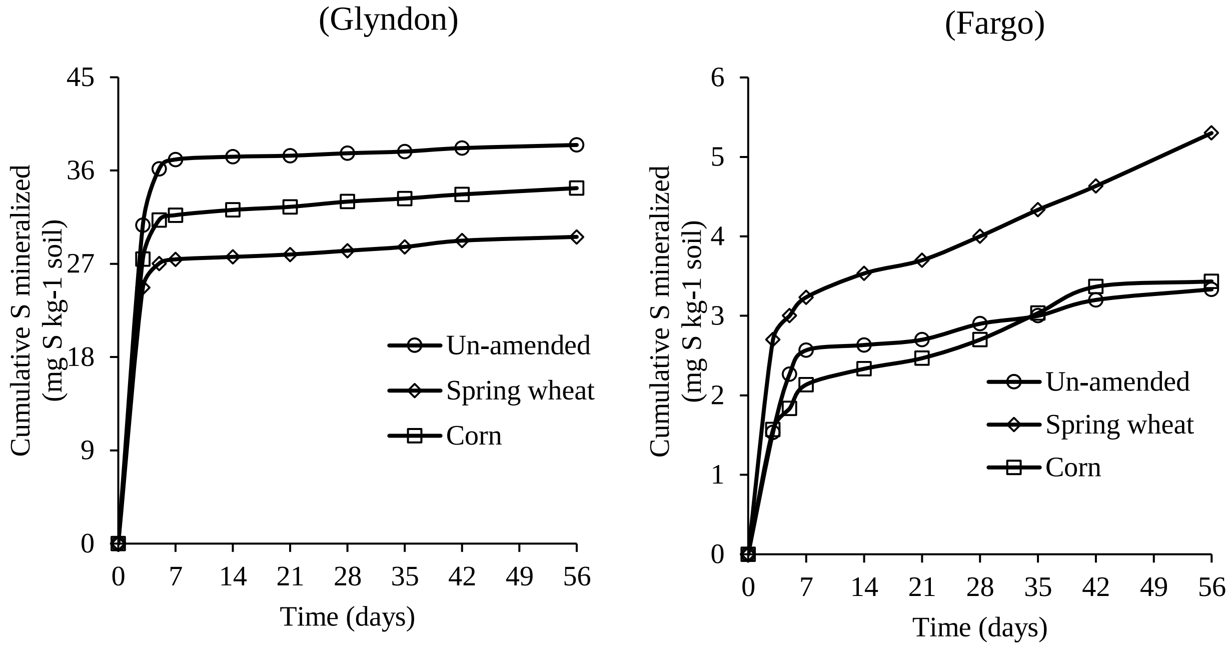

Fig. 6. Cumulative S mineralized in soils amended with crop residues after the 56-d incubation period for Glyndon and Fargo soils.

Table 10. Cumulative S mineralized (mg kg^{-1}) percent of added S mineralized, mineralization potential, and rate constant of soils amended with corn and spring wheat residues after the 56-d incubation period for two contrasting soils.

Soil	Treatment	Cumulative mineralized S (mg kg^{-1})	Percent of added S mineralized[a]	Mineralization Potential (S_0) (mg kg^{-1})	Rate Constant (k_2) (d^{-1})
Glyndon	Un-amended	38.5 (2.90)[b]	-	37.8 (2.43)	0.57
	Corn	34.3 (7.42)	-0.506[c]	33.0 (5.88)	0.58
	Spring wheat	29.6 (3.91)	-0.685	28.4 (3.33)	0.70
Fargo	Un-amended	3.33 (0.40)	-	3.00 (0.36)	0.26
	Corn	3.43 (0.88)	0.012	2.99 (0.51)	0.18
	Spring wheat	5.30 (0.78)	0.152	4.33 (0.72)	0.24

[a] $\frac{(\text{Total S mineralized in amended soil} - \text{total S mineralized in umamended soil}) \times 100}{\text{Amount of S added through residues}}$

[b] Parenthesis include standard deviation

[c] Negative values indicate net S immobilization

The cumulative mineralized S was higher in Glyndon soils as compared to the Fargo soils. Tabatabai and Chae, (1991) found that the S mineralization during a 20-wk incubation period depends on the soil used. Less mineralization occurs in soil more in clay content as clay can protect some of the more easily decomposable organic compounds from rapid microbial breakdown through encrustation and entrapment (Paul and vanVeen, 1978; Anderson, 1979; Tisdall and Oades, 1982). Thus, the higher clay content in the Fargo soil has limited the decomposition of crop residues.

Mineralization is an essential source to fulfill plant S needs. However, in our study, S immobilization demonstrated that crop residues can tie up available S and could result in S deficiency in crops. Further, residues with wide C/N and C/S ratios requires a long period to release the immobilized N and S in soils (Singh et al., 2006), which makes it difficult for short period corn varieties to get S for plant uptake. Therefore, the addition of another S source is essential to initiate the decomposition, especially in the North Dakota region, where cool temperature slows the mineralization of organic substrates.

3.4.6. Sulfur mineralization constants

The potentially mineralizable S (S_0) and rate constants (k_2) are presented in Table 10. Values of S_0 ranged from 28.4 to 37.8 mg kg^{-1} and 2.99 to 4.33 mg kg^{-1} for the Glyndon and Fargo soils, respectively. The rate constant values varied from 0.57 to 0.70 day^{-1} for the Glyndon soils and 0.18 to 0.26 day^{-1} for the Fargo soil. With the Glyndon soil, the S_0 values of the un-amended soil are higher than treated soil. This is similar to a previous study by Islam and Dick, (2008) showing that S_0 of un-amended soils were much greater than those treated with rice straw and pea vines.

In other studies, Reddy et al., (2002) found that S_0 values lie between 16.6 to 32.6 mg kg^{-1} for soils treated with organic materials. Pirela and Tabatabai, (1988) reported that S_0 values calculated by using an exponential equation range from 5 to 44 mg kg^{-1} for Iowa soils. Our Glyndon soil S_0 values lie in this range but the Fargo soil S_0 were below this range. Our calculated S_0 represents 5.7 to 7.75 % of initial soil S for the Glyndon soil that is in line with the findings of Pirela and Tabatabai, (1988), who determined that between 2.4 to 17.5 % of total S was mineralized when soil samples were incubated at 30°C for 98 days.

3.4.7. Relationship between S mineralization and residue properties

Statistical analysis showed that cumulative S mineralized was not significantly correlated with residue properties at $p \leq 0.05$ and $p \leq 0.1$ in Glyndon soils (Table 11). Significant correlations were noticed only when the Fargo soil was considered.

With the Fargo soil, the cumulative mineralized S was highly correlated with cumulative mineralized N and residue S. Pirela and Tabatabai, (1988) reported a significant correlation between cumulative N and S mineralized in a study conducted at 20°C for 14 weeks. A significant negative relationship between cumulative S mineralized and C/S ratio of crop residues was observed in this soil. Earlier, researchers have reported the negative relationship between S mineralization and C/S ratio of different organic materials such as green manure (Tabatabai and Chae, 1991; Reddy et al., 2002), plant material (Eriksen, 2005) and farmyard manure (Tabatabai and Chae, 1991; Reddy et al., 2002).

Table 11. Pearson correlation coefficients between cumulative S mineralized and crop residue properties for Glyndon and Fargo soils.

Site	Parameters	C/N	C/S	N/S	Total C	Total N	Total S
Glyndon	Cumulative S	ns	ns	ns	ns	ns	ns
Fargo	Cumulative S	***	***	***	***	***	***

*** Significant at the 0.1 probability level.
ns, non-significant.

3.5. Conclusion

Addition of crop residues resulted in N and S immobilization in treated soils (except for spring wheat with Fargo soils), indicating that N and S were limiting factors for microbial growth. No clear correlation was found between N and S mineralization and residue properties at both sites. Wide C/N and C/S ratios in the residues and absence of another N and S source resulted in the immobilization. Higher C/N and C/S ratios as well as low N and S in the residues indicates that in the current high yielding crop varieties most of the soil available nutrients are removed with the grain and only small portion is left in the residues. The N and S immobilization can be a concern for growers as they rely on nutrient mineralization from crop residues for nutrient availability during the time of greatest growth of a corn crop; especially, when extremely short season varieties are grown.

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4. GENERAL SUMMARY AND CONCLUSIONS

Corn response to sulfur application rates varied with soil and weather conditions. None of the measured parameters (soil and plant) could correlate well with the corn yield. A response was observed in soils that were high in organic matter whereas no response was noticed in low organic matter soils. A clear picture could have drawn if all S sources and losses were considered. In our study, we did not look at S from mineralization, groundwater, and rainfall. It might be useful if S mineralization was recorded in the field and S up to 60-cm depth was measured. In addition, this study has only two years data. It would be beneficial to conduct the same experiment for some more years with diverse weather conditions to get a concrete conclusion.

In our second experiment, we noticed immobilization with the crop residue additions that showed that residues were low in nutrients and all the available nutrients were tied up in the soil microorganisms. We did not look at microbial population in all the amended and un-amended soils. It might be useful to study the microorganisms involved in mineralization-immobilization process in all soils. Some mineralization could be expected if this experiment was conducted for more months because there was a chance of nutrient release that were tied up in the soil microorganisms. Overall, our 56-d study indicated that crop residue additions early in the growing season might have a negative impact on plant available nutrients due to immobilization of N and S.

APPENDIX A. TABLES

Table A1. Taxonomic classification of ten experimental sites selected in 2016-2017 growing season.

	Sites	Soil series	Taxonomic classification
1	Absaraka	Glyndon	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
2	Ada I	Wheatville	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Aeric Calciaquolls
3	Downer I	Elmville	Coarse-loamy over clayey, mixed over smectitic, superactive, frigid Aeric Calciaquolls
4	Gardner	Fargo	Fine, smectitic, frigid Typic Epiaquerts
5	Walcott I	Fargo	Fine, smectitic, frigid Typic Epiaquerts
6	Ada II	Augsburg	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Typic Calciaquolls
7	Amenia	Glyndon-Tiffany	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
8	Casselton	Bearden	Fine-silty, mixed, superactive, frigid Aeric Calciaquolls
9	Downer II	Lamoure	Fine-silty, mixed, superactive, calcareous, frigid Cumulic Endoaquolls
10	Walcott II	Wheatville	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Aeric Calciaquolls

APPENDIX B. PICTURES

Fig. B1. Sulfur deficiency in Absaraka (site 1) in 2016 growing season.

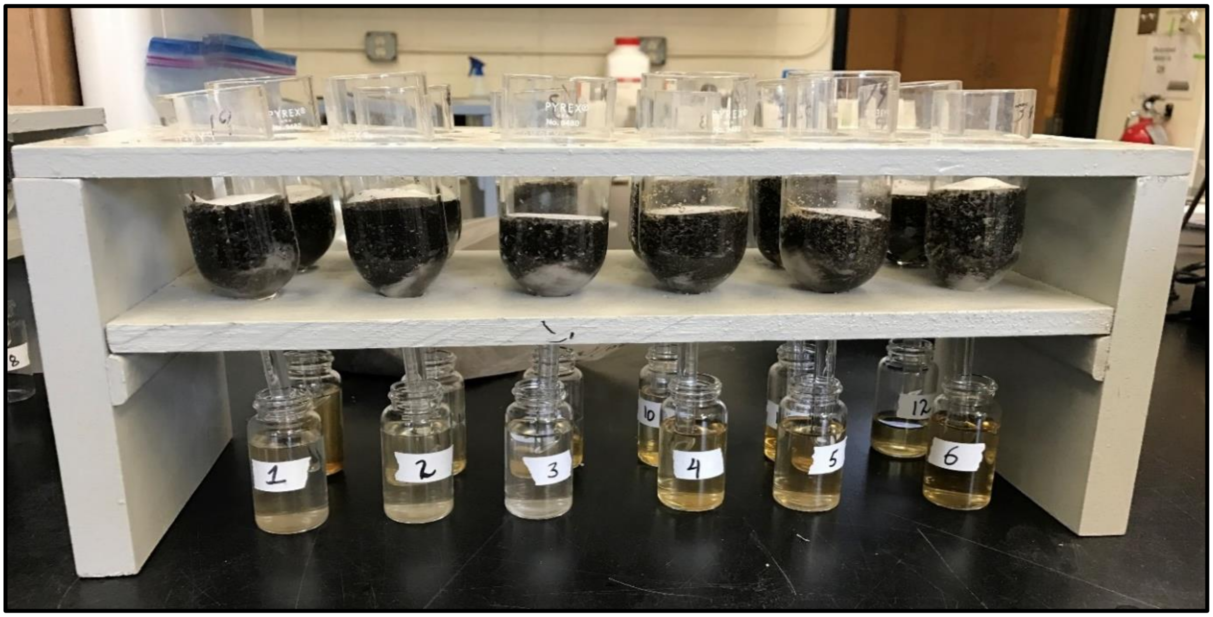

Fig. B2. Experimental setup for N and S mineralization in laboratory.