2021

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Received: 10 September 2020 | Accepted: 30 October 2020

DOI: 10.1111/gcbb.12784

ORIGINAL RESEARCH

GCB-BIOENERGY BIOPRODUCTS FOR A SUSTAINABLE BIOECONOMY Open Access WILEY

Responses of soil surface greenhouse gas emissions to nitrogen and sulfur fertilizer rates to *Brassica carinata* grown as a bio-jet fuel

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Funding information
South Dakota Oilseeds Initiative; USDA-NIFA, Grant/Award Number: 2014-38502-22598

Abstract

Carinata (*Brassica carinata* A. Braun), a non-food oilseed crop and an alternative bio-jet fuel feedstock, has received attention for its potential as a low-input option for production in the semi-arid region of the Northern Great Plains of the United States. Research addressing the impacts of nitrogen (N) and sulfur (S) fertilizers on soils and greenhouse gas (GHG; CO_2, N_2O, and CH_4) emissions from carinata production are limited. Thus, objective of this study was to evaluate the impact of different rates of N and S fertilizers applied to carinata on soil properties and GHG emissions. Field experiments were conducted in 2017 and 2018 to assess the response of carinata to four N (56, 84, 112, and 140 kg N ha^{-1}) and three S (0, 22, and 45 kg S ha^{-1}) rates. Soil samples were collected at crop harvest to measure soil properties; however, soil surface GHG fluxes were measured during 2017 and 2018 growing seasons using static chamber method. Data showed that application of N fertilizer increased soil EC, soil organic carbon (SOC), stable C, and labile N. However, sulfur fertilizer decreased SOC, labile N, and soil inorganic N contents. Results from GHG fluxes showed that higher rates of N fertilizer application increased the soil CO_2 and N_2O emissions, whereas the S fertilizer did not impact these fluxes. This study concludes that S and N fertilizers application to carinata crop affected soil properties, and higher rates of N fertilizer increased the GHG emissions. Therefore, N fertilizer application rate needs to be optimized to mitigate GHG emission for carinata production.

KEYWORDS

biofuel crops, carinata, greenhouse gas, nitrogen rates, soil properties, sulfur fertilizer

1 | INTRODUCTION

Bioenergy has potential to reduce greenhouse gas (GHG) emissions through sustainable resource development and the use of efficient bioenergy systems (Chum et al., 2011). One of the important components for this system is liquid biofuel. Most common biofuel includes bioethanol derived from corn (*Zea mays* L.), sugar beet (*Beta vulgaris* L.), and sugarcane (*Saccharum officinarum* L.), which is mixed with gasoline, whereas oilseed crops-based biofuel, soybean (*Glycine max* L. Merr.), consumes high energy due to short hydrocarbon chains, to produce fuel (Perlack, 2005). Carinata (*Brassica* *carinata* A. Braun), a non-food biofuel crop, has been the focus of much research attention because it can be used as an alternative source of biofuel production (Cardone et al., 2003), which has the potential to reduce global warming through producing low GHG fuel (Agrisoma-USA, 2020). Carinata is originated from the Ethiopian Highlands and is commonly known as Ethiopian mustard (Warwick et al., 2009). Carinata has a mean oil content of 41.7% and a protein content of 30%, depending on the variety and environmental conditions (Hossain et al., 2019). The seed contains long-chain unsaturated fatty acids suitable for the production of bio-jet fuel, lubricants, and bioplastics (Taylor et al., 2010). Carinata is heat and drought-tolerant (Agrisoma-USA, 2020), which makes it an ideal crop for production in semi-arid environments, including central and western South Dakota (SD). Moreover, carinata is also an excellent rotational crop for cereal-based rotations, enhancing overall crop productivity and soil health (Wright, 2017).

Similar to other crops, oilseed crops require N, P, K, and S fertilizers, among which N and S fertilizers are crucial for vegetative and reproductive growth (Abdallah et al., 2010; CFIA, 2017). The current research has less focus on P and K fertilizers as the soil has adequate amount of these nutrients available in the given area. *Brassica* crops have high sulfur demand for the synthesis of sulfur containing compound, glucosinolate, and amino acids, methionine and cysteine, that determines the oilseed quality (Walker & Booth, 2003). Oilseed crops require balanced plant nutrition for enhanced seed and oil production (Ma et al., 2019). The growth and seed yield of carinata were increased with N and S application in India (Verma et al., 2018). A recent study in SD reported that inorganic N fertilizer requirements for carinata are lower compared to the cereal crops (Osborne et al., 2019), which can reduce the greenhouse gas (GHG) emissions (Zhong et al., 2016).

The application of inorganic N fertilizers is important for plant and soil health; however, repeated application of high rates of N fertilizers can affect soil health (Singh, 2018). Growing carinata can help to sustain soil health through low input demand, improvement in soil aggregates, and better carbon sequestering (Agrisoma-USA, 2019). Nitrogen fertilizer can improve soil organic carbon (SOC) content through increased microbial activity (Palmer et al., 2017). A number of studies have found that the long-term application of N fertilizers has significantly improved SOC (Lugato et al., 2010; Zhou et al., 2013). Biomass yield and root growth were improved by the application of inorganic N fertilizers, resulting in improved root activity and accelerated organic C accumulation (Liu et al., 2013). However, a study conducted in SD by Li et al. (2019) reported that increasing N fertilization rates from 0 to 84 kg N ha^{-1} in a carinata crop grown on fine-silt soil did not affect SOC compared to the control. A study conducted in a canola field reported an increase in SOC with the application of N fertilizers due to increased crop residue (Kazemeini et al., 2010). However, a study of mustard (*Brassica juncea* L.) showed increased biomass yield with the application of N fertilizers but unaffected SOC (Prasad et al., 2018). Application of N fertilizer is necessary to replenish the depleted N from soil resulting from plant uptake, leaching or evaporation loss. A study in canola showed that the field receiving higher rates of N fertilizer significantly increased soil mineral N (Herath et al., 2017). Not many studies reported the impact of S fertilizers on SOC under oilseed crops; however, a study in Canada reported a 2–51% reduction in microbial biomass C with the application of elemental S fertilizer in grass field (Gupta et al., 1988). Studies have shown that the application of S fertilizer can increase soil acidity (Fageria et al., 2010; Kissel et al., 2020). However, in contrast, Wiedenfeld (2011) reported that an increase in salinity level was directly correlated with the increasing rate of S fertilizer. Application of ammonium sulfate can result in increased salinity because of higher salt index (3.25) of this fertilizer (Bunt, 1988).

Application of high rates of fertilizers can also cause accumulation and concentration of mineral salts that lead to soil compaction and resistance to root penetration (Massah & Azadegan, 2016). However, inorganic fertilizers (e.g., N, P, and K) were found to have no impact on soil bulk density and SOC content in long-term studies including corn and wheat (Zhou et al., 2013, 2017). Our recent study on the impact of N fertilizers on carinata and camelina (*Camelina sativa* L.) found that there was no significant influence of N fertilizers on SOC (Li et al., 2019). Long-term inorganic N fertilizer application in corn reduced the soil microbial activity and decreased SOC, whereas short-term application of inorganic N fertilizer had limited effects on soil microbial activities (Fauci & Dick, 1994).

Greenhouse gas emissions have an important role in regulating the earth's surface temperature (Zell, 2010); however, higher emissions can make the planet warmer. The United States Environmental Protection Agency (USEPA) (EPA, 2019) reported that agriculture is responsible for 9% of the total GHG emissions in the United States in 2017. Nitrogen fertilizer is one of the main sources of GHG emissions by influencing the processes of microbial decomposition and root respiration (Kim et al., 2014; Li, Watson, et al., 2013; Li et al., 2019; Mbonimpa et al., 2015; Ozlu & Kumar, 2018). There are few data examining the effect of S fertilizer on GHG emissions from oilseed crops. Studies in Canada (Gupta et al., 1988) and China (Wang et al., 2008) reported that the application of S fertilizer reduces microbial biomass and soil pH from grass fields, which can impact the soil GHG emissions. Our previous study by Li et al. (2019) found that increasing N fertilizer application (0–84 kg N ha^{-1}) in carinata resulted in higher N_2O emissions. This research further extended the study of Li et al. (2019) by evaluating the impact of N (>84 kg N ha^{-1}) and S fertilizer applications to carinata on GHG emissions. The N rate to carinata did not impact SOC and total N (TN) in our previous study (Li et al., 2019) as changes in these parameters due to management practices occur very slowly (Purakayastha et al., 2008; Zhong et al., 2014). Therefore, the response of labile and stable soil C and N to different N and S fertilizer rates applied to carinata were evaluated in the current study. Specific objective of this study was to assess the impacts of different N and S fertilizer rates applied to carinata on soil properties including soil bulk density (BD), pH, electrical conductivity (EC), SOC, TN, stable and labile C and N, and surface GHG fluxes.

2 | MATERIALS AND METHODS

2.1 | Site description and treatment details

The study was conducted at Aurora Agricultural Experiment Station near Brookings, South Dakota (44º18′35″N, 96º40′15.9″W) during 2017 and 2018. The experiment was established on Brandt series soils characterized by fine silty, super active, frigid calcic hapludolls (Malo, 2003; Soil Survey Staff, 2017). Carinata fits well in rotation with wheat and other small grains. South Dakota has over 800,000 ha (USDA-NASS, 2012) dedicated to wheat production therefore, excellent potential land for carinata production in rotation with wheat. Therefore, the present study was established on winter wheat stubble each year but with different fields used in 2017 and 2018. The average soluble salts at the experimental site in 2017 was 100 μS cm^{-1}. The pH of the soil was found to be acidic (pH = 5.6), and P, K, and S levels were 10 mg kg^{-1}, 141 mg kg^{-1}, and 9.0 kg ha^{-1}, respectively, with 47 g kg^{-1} organic matter. The pH of the soil in 2018 site was 5.7 with 53 g kg^{-1} organic matter, and P and K levels were 21 and 220 mg kg^{-1}, respectively. The planting dates of carinata in 2017 and 2018 were 24 April and 8 May, respectively. Planting was done using a seven-row Hege 500® (Wintersteiger-Austria) at 22 cm row spacing. After crop emergence, Poast (Sethoxydim, BASF, Research Triangle, NC) herbicide was applied at the rate of 2.1 L ha^{-1} 4 weeks after planting to control grassy weeds. Broadleaf weeds were managed by manually removing weeds from each plot as required. Carinata was harvested in August and September in 2017 and 2018, respectively. According to US Climate Data 2020, the 30 year (1980–2010) annual mean precipitation was 617 mm and mean high and low temperature were 12.2°C and 0°C, respectively, whereas the mean precipitation, high and low temperature during the cropping season (April–September) were 496 mm, 22.2°C, and 9.6°C, respectively.

The experimental plots were 1.62 m wide by 9.14 m long, arranged in a randomized complete block design (RCBD) with 12 treatments and three replications. Study treatments included four different rates of N fertilizer: 56, 84, 112, and 140 kg N ha^{-1}, and three different rates of S fertilizer: 0 (control), 22, and 45 kg S ha^{-1} arranged in a factorial design to make 12 treatments within each replication for both years. A previous study by Osborne et al. (2019) has determined the optimum N rate for carinata is between 60 and 81 kg N ha^{-1}; thus, we used the lowest N rate of 56 kg ha^{-1} and increased the rate at the interval of 28 up to 140 kg N ha^{-1}. The optimum S fertilizer required to canola, crop similar to carinata, was around 20 kg S ha^{-1} (Jackson, 2000); thus, based on this study, we selected three different S fertilizer rates. Both urea (46-0-0) and ammonium sulfate (21-0-0-24) were broadcast in two-equal spilt doses: first half immediately after planting (24 April 2017 and 8 May 2018) and the next at bolting stage (13 June 2017 and 22 June 2018).

2.2 | Soil surface GHG monitoring

Soil GHG fluxes were monitored for the whole growing season of carinata in 2017 and 2018. Nine treatments (a factorial combination of 56, 112, and 140 kg N ha^{-1} and 0, 22, and 45 kg S ha^{-1}) in three replications were selected for monitoring the GHG emissions. The whole experimental plots were not selected for gas sampling because of resources and time limitations and the study was focused on determining treatment differences; thus, we selected the lowest N rate and two highest N rate treatments. Polyvinyl chloride (PVC) static chambers (25 cm diameter and 15 cm height) were installed in each plot to monitor soil surface GHG fluxes, according to the method of Parkin and Venterea (2010). Gas samples were taken once or twice a week depending on weather conditions from June to August in 2017, and from May to August in 2018. During the GHG sampling, a lid (10 cm height with ports for gas sampling) was placed on top of the anchor. Gas samples were collected three times (0, 20, and 40 min) over an hour period by inserting a needle attached to a 10 ml syringe into the port and transferring the gas to pre-evacuated 10 ml vials. Gas chromatograph was used to measure the concentrations of CO_2, N_2O, and CH_4 for each sample. Soil GHG fluxes were calculated as the change in headspace gas concentration over time within the enclosed chamber volume (Hutchinson & Mosier, 1981; Ussiri & Lal, 2009). During each gas-sampling event, soil temperature at the 0–5 cm depth was measured with a thermometer (Taylor 14769 Digital 0.7" Lcd Folding Thermometer). The volumetric soil moisture content (%) at 0–5 cm depth was also measured at the time of gas sampling using a thetaprobe moisture sensor (Delta-T-Devices).

2.3 | Soil sampling

Soil samples were collected from the experimental site in August 2017 and November 2018 at 0–5, 5–15, 15–30, and 30–45 cm depths using a soil probe (3.2 cm diam.). For the analysis of pH and electric conductivity (EC), soil samples were air-dried and ground to pass through a 2 mm sieve, and pH and EC (1:2.5 soil/water) were determined as described by McLean (1982). Total C and N were measured by dry combustion using TruSpec carbon–hydrogen–nitrogen analyzer (LECO Corporation). Soil samples had low pH (≤6); therefore, measured TC was considered equal to SOC (Guo et al., 2016). Soil C and N fractions were analyzed using cold water extraction (for labile C and N) and hot water extraction (for stable C and N) methods (Ghani et al., 2003). A TOC-L analyzer (Shimadzu Corporation, model-TNM-L-ROHS) was used to determine the cold-water and hot-water C (CWC and HWC) and N (CWN and HWN) fractions. Soil bulk density was determined using the core method (Grossman & Reinsch, 2002).

2.4 | Statistical analysis

The impacts of N and S fertilizers applied to carinata on selected soil parameters in 2017 and 2018 were analyzed using PROC MIXED method. In this analysis, the year, N fertilizer, S fertilizer, and their interactions were considered fixed effects and replication as a random effect. No significant interactions were found; therefore, only main effects are discussed in this paper. There were no significant differences among soil properties between depths; hence, average values from 0 to 45 cm depth were compared. Statistical comparisons of soil BD, pH, EC, SOC, TN, C-fractions, and N-fractions between N and S fertilizers rates were obtained using pairwise differences method (adjusted by Tukey) in a mixed model approach using GLIMMIX procedure. The repeated measures analysis for comparing the soil CO_2, CH_4, and N_2O fluxes under different N and S rates was conducted using PROC MIXED, with N and S fertilizers and year as fixed effects, replication as a random effect, and date of gas sampling as a repeated measure variable. All statistical analyses were conducted using SAS 9.4 at a significance level of $\alpha = 0.05$.

3 | RESULTS

3.1 | Weather conditions, soil moisture, and temperature

Air temperature and rainfall data throughout the crop growing period at the site in 2017 and 2018 are shown in Figure 1. During the critical crop growth period (June–July), temperature often exceeded 25°C in both years. In 2017, the experimental site received a total rainfall of 377.2 mm over the crop-growing season (April–August); most of the precipitation was received in July and August (Figure 1). The early days of June 2017 did not receive significant rainfall but were followed by scattered rainfall mid-month. Unlike 2017, the 2018 year was a wet year where the total amount of rainfall during the growing period was 442.9 mm; on a single day in mid-July, the total rainfall was 150 mm (Figure 1). Soil temperature and moisture for different N and S rates during the crop growing period are presented in Figure 2. The soil temperature trend in the 2017 growing season was similar to that of 2018 under N and S fertilizer rates (Figure 2a,b). Soil moisture was lower in the 2017 (average 16.9%) season compared to the 2018 (average 18.4%) for N and S fertilizer rates ($p \leq 0.05$, Figure 2c,d). Soil temperature and moisture (Figure 2) were not affected by N and S fertilizer rates in 2017 and 2018 (averaged across measurement dates in each year, $p > 0.05$).

3.2 | Soil properties

Data showed that none of the studied treatments significantly influenced soil bulk density in either year (Tables 1 and 2). The mean soil pH was acidic, but was not influenced by

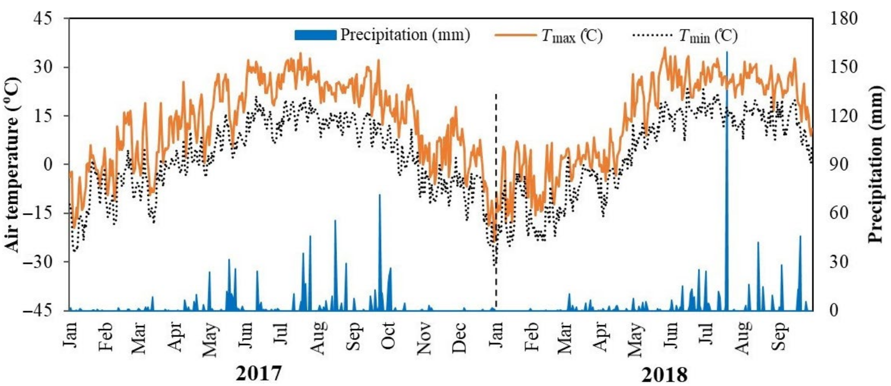

FIGURE 1 Daily maximum and minimum air temperature and precipitation in 2017 and 2018 for the study site. Orange line represents the maximum air temperature, dotted line represents the minimum air temperature, and blue bar represents the precipitation

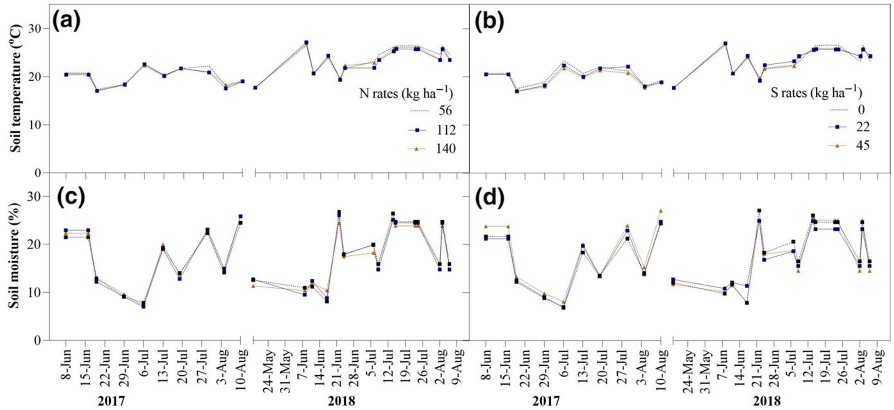

FIGURE 2 Average soil temperature of plots applied with different (a) N rates, and (b) S rates and average soil moisture of plots applied with different (c) N rates and (d) S rates over the observed days in 2017 and 2018

TABLE 1 Means of soil bulk density (BD), pH, electrical conductivity (EC), soil organic carbon (SOC), total nitrogen (TN), and labile and stable C and N as influenced by different N and S fertilizers rates to carinata for the 0–45 cm depth in 2017

	BD (g cm^{-3})	pH	EC (µS cm^{-1})	SOC (g kg^{-1})	TN (g kg^{-1})	Labile C (mg kg^{-1})	Stable C (mg kg^{-1})	Labile N (mg kg^{-1})	Stable N (mg kg^{-1})
N rates (kg ha^{-1})									
56	1.4†	5.4	322.4^{b}	24.5^{b}	2.7	288.6	936.3^{b}	50.0^{b}	30.5
84	1.4	5.4	319.2^{b}	25.1^{ab}	2.7	307.9	992.5^{ab}	54.4^{b}	33.1
112	1.4	5.4	368.6^{a}	26.0^{a}	2.7	311.1	1071.9^{a}	90.0^{a}	35.8
140	1.4	5.3	370.0^{a}	26.0^{a}	2.9	297.1	1020.4^{a}	67.5^{ab}	34.1
	Analysis of variance ($p > F$)								
	0.306	0.137	0.009	0.041	0.058	0.573	0.012	0.013	0.114
S rates (kg ha^{-1})									
0	1.4†	5.3^{b}	369.2^{a}	25.2	2.7	286.1	1016.2	63.1	33.4
22	1.4	5.5^{a}	329.9^{b}	25.7	2.7	306.7	967.6	68.5	32.6
45	1.4	5.5^{a}	336.1^{b}	25.3	2.8	308.2	1028.2	62.2	33.9
	Analysis of variance ($p > F$)								
	0.363	0.016	0.007	0.634	0.241	0.535	0.103	0.991	0.487

[†]Mean values followed by different lowercase letters within the column represent significant differences due to the treatments at $p \leq 0.05$. Values are the average values for 0–5, 5–15, 15–30, and 30–45 cm depth soil samples as there was no significant difference between the depths.

different N fertilizer treatments in either year (Tables 1 and 2). However, soils with S fertilizer applied at the rate of 22 and 45 kg S ha^{-1} in 2017 had significantly higher pH values than 0 kg S ha^{-1} (Table 1). Application of S fertilizer in 2018 did not affect pH (Table 2). Mean EC at 112 and 140 kg N ha^{-1} in 2017 were significantly higher than the 56 and 84 kg N ha^{-1} (Table 1). While the application of 22 and 45 kg S ha^{-1} reduced the EC by 12% and 10%, respectively, compared to S control soils in 2017 (Table 1), and S control plot had significantly lower EC compared to higher rates in 2018 (Table 2). For SOC, 140 and 112 kg N ha^{-1} increased SOC more than the 56 kg N ha^{-1}; however, 84 kg N ha^{-1} had a similar concentration compared to all other rates in 2017 (Table 1). As seen in Table 2, N fertilizer rates did not show any significant impact on SOC in 2018. Sulfur fertilizer rates showed a significant impact only in 2018, when the application of 22 and 45 kg S ha^{-1} had 3.2% and 1.8% lower SOC than the S control soils, respectively (Table 2). Nitrogen and S fertilizers did not influence total soil N in either year (Tables 1 and 2). Thus, total N results are not discussed here. Data on C and N pools showed significant impact of N fertilizers only on stable C and labile N in 2017, whereas these parameters were not influenced in 2018 (Tables 1 and 2). Stable C in 2017 was higher under 140 and112 kg N ha^{-1} than 56 kg N ha^{-1} but similar to 84 kg N ha^{-1} (Table 1). Labile N in 2017 was higher under 112 kg N ha^{-1} compared to 84 and 56 kg N ha^{-1} (Table 1). Data on C and N pool were non-significant to S fertilizers in 2017 (Table 1). In 2018, however, soil in the 45 kg S ha^{-1} had 17% lower concentration of labile N compared to S control soil, which was similar to 22 kg S ha^{-1} (Table 2).

TABLE 2 Means of soil bulk density (BD), pH, electrical conductivity (EC), soil organic carbon (SOC), total nitrogen (TN), and labile and stable C and N as influenced by different N and S fertilizers rates to carinata for the 0–45 cm depth in 2018

	BD (g cm^{-3})	pH	EC (μS cm^{-1})	SOC (g kg^{-1})	TN (g kg^{-1})	Labile C (mg kg^{-1})	Stable C (mg kg^{-1})	Labile N (mg kg^{-1})	Stable N (mg kg^{-1})
N rates (kg ha^{-1})									
56	1.4	5.6	123.2	28.6	3.1	105.5	420.7	21.9	43.2
84	1.4	5.7	120.3	29.0	3.1	110.8	436.2	23.8	46.6
112	1.5	5.6	126.5	28.3	3.0	106.5	418.8	23.3	43.7
140	1.5	5.7	125.9	28.4	3.1	100.8	394.6	23.9	41.9
	Analysis of variance ($p > F$)								
	0.804	0.401	0.656	0.266	0.478	0.167	0.180	0.427	0.173
S rates (kg ha^{-1})									
0	1.5	5.6	116.0[b†]	29.0[a]	3.1	108.6	424.4	24.2[a]	45.4
22	1.4	5.7	129.7[a]	28.1[b]	3.1	108.9	418.6	25.5[a]	44.6
45	1.5	5.6	126.6[a]	28.5[b]	3.0	100.7	411.6	20.1[b]	41.9
	Analysis of variance ($p > F$)								
	0.908	0.281	0.027	0.011	0.254	0.091	0.531	0.001	0.106

[†]Mean values followed by different lowercase letters within the column for N and S rates represent significant differences due to the treatments at $p \leq 0.05$. Values are the average values for 0–5, 5–15, 15–30, and 30–45 cm depth soil samples as there was no significant difference between the depths.

3.3 | Soil GHG fluxes

Data on daily mean soil GHG fluxes from carinata crop managed with different N and S fertilizers rates over the observed days in 2017 and 2018 are presented in Figure 3. For N fertilizer, CO_2 peaks were observed on 16 June and 13 July in 2017 (Figure 3). No significant differences on CO_2 fluxes were observed among N fertilizer rates during these two peaks in this year ($p > 0.05$, analysis not shown in the figure). The largest difference among N fertilizer rates in CO_2 fluxes was observed on 6 July 2017, with 56 kg N ha^{-1} recording lower CO_2 flux (16.28 kg CO_2-C ha^{-1} day^{-1}) than 140 kg N ha^{-1} (24.04 kg CO_2-C ha^{-1} day^{-1}) and 112 kg N ha^{-1} (21.12 kg CO_2-C ha^{-1} day^{-1}; $p < 0.001$, Figure 3). In 2018, the highest CO_2 fluxes were observed on 24 June and 6 July, with 140 and 112 kg N ha^{-1} recording higher CO_2 fluxes than 56 kg N ha^{-1} ($p < 0.001$, Figure 3). The peaks of CO_2 flux under S fertilizer rates were observed on 16 June and 13 July in 2017; however, no significant differences on CO_2 fluxes were observed among S fertilizer rates during these two peaks ($p > 0.05$). In 2018, soil CO_2 fluxes increased from May to the beginning of July and decreased thereafter under all S fertilizer rates (Figure 3). The fluxes of CO_2 were similar among S fertilizer rates at all sampling dates in 2018 ($p > 0.05$).

For N_2O fluxes, the peaks in 2017 were recorded on 16 June, 19 and 29 July, with 112 and 140 kg N ha^{-1} recording higher N_2O than 56 kg N ha^{-1} in all peaks ($p < 0.001$, Figure 3). In 2018, the highest daily peak was seen on 24 June (Figure 3). Again, 112 and 140 kg N ha^{-1} emitted higher N_2O than 56 kg N ha^{-1} during this peak ($p < 0.001$, Figure 3). The peaks of N_2O fluxes were also observed under S fertilizer treatment on 16 June, 19 and 29 July 2017, and 24 June, 6 and 24 July 2018, but no significant differences on N_2O flux among treatments were recorded in either year at any sampling dates ($p > 0.05$, Figure 3). The trend of the CH_4 fluxes under N and S fertilizers varied on the sampling dates over the 2 years (Figure 3). There were no clear seasonal patterns over the 2 years under all treatments (Figure 3). However, no significant effects of N and S fertilizer rates on the daily CH_4 fluxes were observed during the sampling dates for both years ($p > 0.05$).

Data on the seasonal means of CO_2, N_2O and CH_4 fluxes as influenced by different N and S fertilizer treatments are presented in Table 3. In 2017, application of N fertilizer rates significantly influenced CO_2 flux, with 140 kg N ha^{-1} recording higher CO_2 flux than 56 kg N ha^{-1}; however, the flux at 112 kg N ha^{-1} was similar with that of both 56 and 140 kg N ha^{-1} (Table 3). No significant differences on CO_2 fluxes due to N fertilizer rates were observed in 2018 (Table 3). The fluxes of N_2O were lower in the plots with 56 kg N ha^{-1} than 112 and 140 kg N ha^{-1} in 2017 (Table 3). Like CO_2 flux, no significant differences in N_2O fluxes due to N fertilizer rates were observed in 2018 (Table 3). Methane fluxes were unaffected by N and fertilizer rates in both years (Table 3).

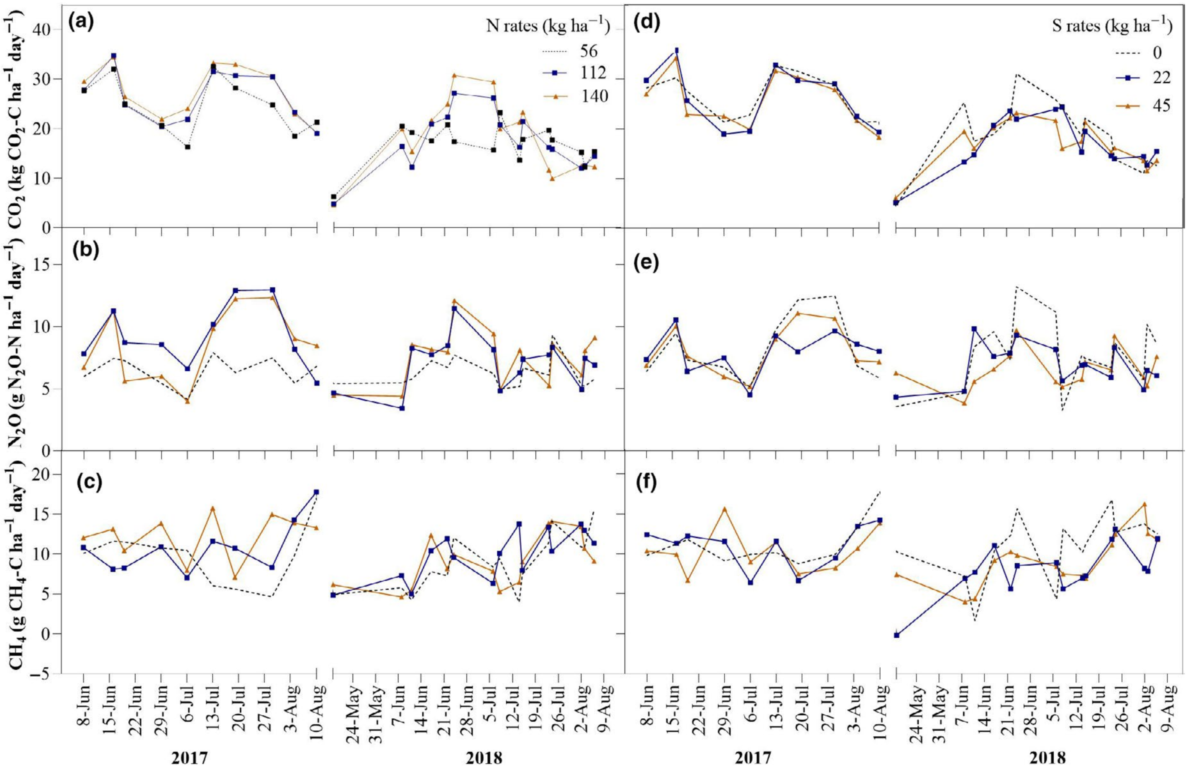

FIGURE 3 Daily mean soil CO_2, N_2O, and CH_4 fluxes from carinata crop managed with different N fertilizer rates (a, b, c, respectively) and S fertilizer rates (d, e, f, respectively) over the observed days in 2017 and 2018

TABLE 3 Means of CO_2, N_2O, and CH_4 fluxes as influenced by different N and S fertilizer rates to carinata in 2017 and 2018

	2017			2018		
	CO_2 (kg C ha^{-1} day^{-1})	N_2O (g N ha^{-1} day^{-1})	CH_4 (g C ha^{-1} day^{-1})	CO_2 (kg C ha^{-1} day^{-1})	N_2O (g N ha^{-1} day^{-1})	CH_4 (g C ha^{-1} day^{-1})
N rates (kg ha^{-1})						
56	24.8 (±5.2)[b†]	6.5 (±1.2)[b]	9.6 (±3.4)	16.8 (±3.9)	6.3 (±1.1)	8.9 (±3.4)
112	26.4 (±5.1)[ab]	9.4 (±2.4)[a]	10.4 (±3.1)	17.3 (±5.7)	7.1 (±1.9)	9.9 (±2.9)
140	27.2 (±5.1)[a]	8.5 (±2.8)[a]	12.5 (±2.7)	18.1 (±7.2)	7.6 (±2.0)	9.1 (±3.1)
	Analysis of variance ($p > F$)					
	0.015	<0.001	0.118	0.954	0.109	0.623
S rates (kg ha^{-1})						
0	26.4 (±4.2)	8.3 (±2.4)	11.3 (±2.6)	17.7 (±6.1)	7.9 (±2.9)	10.9 (±3.7)
22	26.3 (±5.7)	8.0 (±1.6)	11.0 (±2.5)	16.9 (±5.1)	6.8 (±1.5)	8.1 (±3.1)
45	25.7 (±5.1)	8.0 (±1.9)	10.1 (±2.6)	16.9 (±4.5)	6.5 (±1.5)	9.3 (±3.2)
	Analysis of variance ($p > F$)					
	0.487	0.893	0.803	0.708	0.054	0.060

Note: Standard error values (±) are shown in the parentheses.

[†]Mean values followed by different lowercase letters within the column for N and S rates represent significant differences due to the treatments at $p \leq 0.05$.

4 | DISCUSSION

4.1 | Soil moisture and temperature

Increasing the N rate from 56 to 140 kg N ha^{-1} in this study did not affect soil moisture and temperature. In line with the results of current study, our previous study reported no impact of N fertilizer applications (0–84 kg N ha^{-1}) to carinata did not affect soil temperature and moisture for 3 years which was attributed to the short growing season of the carinata crop (Li et al., 2019). Similarly, nitrogen fertilizer application (0–112 kg ha^{-1}) to switchgrass (*Panicum virgatum* L.) did not affect soil moisture and temperature in South Dakota (Mbonimpa et al., 2015). Conversely, Sainju, Caesar-TonThat, et al. (2012) reported that the N fertilization to malt barley (*Hordeum vulgaris* L.) decreased soil temperature compared to the control (no fertilizer). This difference in the effect of N fertilization rates on soil temperature between our study and Sainju, Caesar-TonThat, et al. (2012) can be attributed to the differences in crops used in each study. No differences on soil moisture and temperature were observed among S fertilizer treatments in this study. The impact of S fertilizer rates on soil moisture and temperature has not been reported in the literature yet. Bulk density can impact the soil pores and hence the soil moisture. In general, a higher bulk density can reduce the soil pores and the moisture. In this study, bulk density was similar under all N and S fertilizer rates in both years (Tables 1 and 2), perhaps explaining why similar soil moisture were recorded among the treatments in this study.

4.2 | Soil properties

Data showed that there was no significant impact of N and S fertilizer rates on soil bulk density, which can be attributed to the short duration of the study (2 years). Long-term application of fertilizers may affect soil bulk density (Blanco-Canqui et al., 2015). Singh et al. (2019) reported that a 10-year application of 112 kg N ha^{-1} to switchgrass decreased soil bulk density compared to the control (no fertilizer). The increased biomass production and residue retention on the soil under high N fertilization can increase SOC and reduce soil bulk density (Wagner et al., 1994). Similar to our findings, a study of maize crop in China Zhong et al. (2014) reported non-significant impacts of different rates of NPK fertilizers including urea on soil porosity and bulk density. Similarly, even a 17-year study on chemical fertilization (N and P fertilizers) in China did not show any significant impact on soil bulk density (Li, Xu, et al., 2013).

Nitrogen fertilizer rate did not affect soil pH in either year. Excess N fertilizer in soil is associated with H^+ ion formation (Barak et al., 1997; Zhang et al., 2017), which was not observed in this study. Bryla et al. (2008) reported ammonium-releasing fertilizers lower the pH gradually, and so their effect might not be detectable in a short period. Similar results for the soil pH in response to different N rates were reported by a study on switchgrass (Mbonimpa et al., 2015). However, the change in pH with S fertilizer application might be due to H^+ ions being released after microbial conversion of ammonium to nitrate (nitrification; Barak et al., 1997). One molecule of ammonium sulfate can release four H^+ ions (IPNI, 2019) in the soil, which lowers the soil pH; however, our results are contradictory to this, which might be due to the release of hydroxide ions in soil during the nutrients uptake by plants. When plants uptake NO_3-N, it equals or exceeds the uptake of potassium, calcium, and magnesium, and leading to proton consumption by plants and thus, hydroxide is released in the soil (Jaillard et al., 2003). Our results were similar to the findings from Wiedenfeld (2011) where the application of S fertilizer increased soil salinity. High soil water content might have limited the nitrification process, which would prevent the release of H^+ ions (Tu et al., 2000), thus exhibiting no effect on soil pH from N and S fertilizer rates.

Increasing N fertilizer rates increased the EC of the soil in 2017, supporting the results reported by Liu et al. (2014). Similarly, our previous study reported that increasing N rates increased the soil EC, which was associated with the increase in the salts due to the fertilizer application (Li et al., 2019). Both fertilizers, urea and ammonium sulfate, serve as the source of N in the soil; higher salt index of ammonium sulfate (3.25) might have increased the salt concentration in the soil. Our result was supported by the findings by Han et al. (2015) and Gandois et al. (2011). The increase in soil EC with higher S fertilizer rate in 2018 may be due to the higher concentration of ammonium sulfate (Bunt, 1988). The addition of ammonium sulfate linearly increased the saturated EC (Bryla et al., 2008). Soil EC increases with the application of S fertilizer (SO_4^-), as reported by other researchers (Hashemimajd et al., 2012; Turan et al., 2013). Soil EC decreased with increasing S fertilizer rates in 2017, which is not common and thus, additional measurements are needed to determine this impact.

The current study found increased SOC with increasing use of N fertilizer. When N is a limiting factor, N fertilizer can impact SOC mineralization through influencing microbial activity and plant biomass (Chen et al., 2014). Sekaran et al. (2019) reported that increasing N rates in soil seeded with switchgrass increased the activity of urease and β-glucosidase enzymes. Other studies reported an increase in SOC with increased N fertilizer rates (Ghimire et al., 2017; Li et al., 2015). Increase in SOC might also have resulted from higher crop biomass and C sequestration (Giacometti et al., 2013). Carinata produced higher biomass under higher N rate (data not shown), which may have added organic carbon to the soil through microbial activity (Seepaul et al., 2016). A study in continuous corn and corn–soybean rotation (Poffenbarger et al., 2017) stated that application of N fertilizer increased SOC until an optimum N rate, above which SOC storage was not affected. Analogous to this study, SOC increased until the application of 112 kg N ha^{-1} and remained constant with further increase in N fertilizer rate in our present study. The application of S fertilizer in 2018 reduced the SOC compared to the S control plots, which might be due to the acidic effect of sulfate in 2017 causing reduced enzyme activity (Lv et al., 2014). The presence of high H^+ ions can inhibit soil microbial activity (Chen et al., 2012). A study in Canada by Gupta et al. (1988) showed that the application of S fertilizer reduced microbial biomass.

A study reported that increase in stable C with higher rates of N fertilizer might be due to the residue decomposition accelerated by higher microbial activity (Alvarez, 2005). Our results of increased stable C with higher N fertilizer rates are in line with the previous study. Water extractable labile N is the source of energy for soil microbial activity (Zhang et al., 2016), which increased with increasing N fertilizer rate in 2017. However, labile N decreased in 2018 with the application of S fertilizer, which might be attributed to the reduced SOC. The non-significant influence on other C and N fractions is analogous to the data reported by Singh et al. (2019) in their study of switchgrass in South Dakota.

4.3 | Daily and seasonal GHG fluxes

The CO_2 fluxes varied with date of sampling, peaked after short precipitation and fertilizer application. Similar findings were also reported by Sainju, Stevens, et al. (2012). Our results showed that the CO_2 fluxes peaked on 16 June in 2017 might probably have resulted from the similar phenomenon as explained by previous studies (Sainju, Stevens, et al., 2012). There were similar activities including increased soil temperature, soil moisture (precipitation from 10 to 14 June), and fertilizers application (13 June) that might have increased the CO_2 peak. Peaks on 13 July, 2017 resulted from precipitation on 12 July and higher soil temperature (Figures 1 and 2). In 2018, the peaks in June and July might have resulted from fertilizers application (22 June) and continuous rainfall from 17 June to 21 June. A number of studies have reported significant effects of soil temperature, soil moisture, and fertilizer application on CO_2 fluxes (Abagandura, Şentürklü, et al., 2019; Davidson et al., 1998; Mbonimpa et al., 2015; Schaufler et al., 2010; Soosaar et al., 2011). Similar to our results, application of N and S increased CO_2 emissions in a study by Hu et al. (2017), which might be due to increased root respiration with the application of exogenous nutrients.

The N_2O peaks in June and July in 2017 were a response to N and S fertilizers, higher temperature, and soil moisture resulting from heavy rainfall (Figures 1 and 2), which may trigger N mineralization and the nitrification/denitrification process, leading to higher N_2O emissions. Similar findings were also reported by Chatskikh and Olesen (2007). Similar to CO_2 fluxes, the N_2O peaks in 2018 were observed on 24 June, probably due to fertilizer application (22 June) and continuous rainfall (17–21 June), resulting in higher soil moisture and temperature (Figures 1 and 2). These conditions may induce the nitrification/denitrification process, resulting in high N_2O emission. Many studies reported loss of applied N because of the increased stimulatory response of N_2O emissions (Drury et al., 2014; Omonode et al., 2011; Pelster et al., 2011). Application of ammonium sulfate releases NH_4^+, which is the substrate for nitrification and the aerobic source of N_2O (Deppe et al., 2016); however, N_2O fluxes often peak under oxygen-limited denitrifying (high soil moisture, fertilization) conditions (Drury et al., 2006; Pelster et al., 2012).

Soil CH_4 fluxes were very low for both years in this study. Several studies have reported low soil CH_4 fluxes under aerobic soil conditions (Abagandura, Şentürklü, et al., 2019; Li et al., 2019). Methane flux was positive on most of the sampling dates in both years, perhaps due to heavy and continuous rainfall in both years (Figure 1), resulting in high soil moisture (Figure 2), which, in turn, may have produced an anaerobic condition. Release or uptake of CH_4 depends on the microbes present in the soil. The anaerobic microbes release CH_4 and increase these fluxes, while aerobic microbes uptake CH_4, and result in negative flux (Abagandura, Chintala, et al., 2019). Higher amounts of crop residues (senesced leaves of carinata) at the maturity stage, in combination with moisture and higher temperature, might have triggered the decomposition process, which is the substrate for CH_4 production (Ding et al., 2004).

The increase in mean seasonal CO_2 flux with increasing rate of N fertilizer in 2017 may be due to higher microbial activity, which increases C and N mineralization, as discussed earlier. The SOC, stable C, and labile N significantly increased with increasing N fertilizer rates, which may cause this increase in CO_2 flux with increasing N fertilizer rate. Similar to our results, Sainju et al. (2008) reported a 14% increase in CO_2 fluxes in N-applied soils compared to the N control soils in North Dakota. The present research results have concurred with many other studies (Hu et al., 2017; Jiang et al., 2010). In contrary, a study by Mbonimpa et al. (2015) reported a reduction in CO_2 fluxes in plots applied with N fertilizer due to the combination of lower SOC, lower porosity, and higher bulk density.

Nitrogen fertilizer increased mean seasonal N_2O flux in 2017, which may be due to N mineralization and the nitrification/denitrification process, depending on the environmental conditions throughout the crop-growing period. Our results of seasonal N_2O fluxes agree with other studies (Drury et al., 2014; Dusenbury et al., 2008). In 2018, there was no significant influence of either fertilizer on CO_2, and N_2O fluxes; higher precipitation might have resulted in nitrate leaching, which could reduce the amount of volatilizing N and, in addition, higher precipitation might have reduced the soil air space by filling soil pores with water. In addition, soil properties were least affected by the fertilizer treatments in 2018. Sulfur fertilizer rates did not affect CO_2 and N_2O fluxes in both years in this study, suggesting that increasing S fertilizer rate from 0 to 45 kg S ha^{-1} in carinata field for 2 years will not increase the flux of these gases under similar conditions.

The lack of effect of N and S fertilization rates on CH_4 fluxes in this study was attributed to the similar soil moisture among treatments. Several studies reported no significant effect of N fertilizer rate on soil CH_4 fluxes compared with the control from bioenergy crops in the dryland cropping system (Abagandura, Chintala, et al., 2019; Li et al., 2019; Mbonimpa et al., 2015). Management practices usually have little effect on CH_4 flux in the upland agricultural soils, as reported in our previous studies (e.g., Abagandura, Şentürklü, et al., 2019; Ozlu & Kumar, 2018).

5 | CONCLUSIONS

This study demonstrated the impact different rates of N and S fertilizers applied to carinata on soil properties and greenhouse gas fluxes in Brookings, South Dakota in 2017 and 2018. The growing season of 2018 was a comparatively wet year with heavy precipitation. Data from this study showed mixed impacts of N and S fertilizers on soil parameters. In 2017, N fertilizer rates significantly increased soil EC, SOC, stable C, and labile N, whereas the S fertilizer rates decreased the soil acidity and EC. In 2018, application of S fertilizer increased soil EC but decreased the SOC. The application of N fertilizer at higher rates significantly increased CO_2 and N_2O emissions only in 2017. No significant influence of S fertilizer application was found on GHG emissions in either year. We can conclude from this study that, in general, sulfur fertilizer at a rate of 0–45 kg S ha^{-1} did not affect GHG fluxes but required for the carinata crop growth. Furthermore, application of N fertilizer did not impact soil properties, however, higher N fertilizer rate (140 kg ha^{-1}) increased soil surface CO_2 and N_2O fluxes. This study suggests that the application of N fertilizer beyond 112 kg N ha^{-1} can increase soil surface GHG emissions, and this N rate with 22 kg S ha^{-1} needed for the carinata growth with minimal GHG emissions.

ACKNOWLEDGEMENTS

The project was funded by the South Dakota Oilseeds Initiative, the North Central Regional Sun Grant Center at South Dakota State University through a grant provided by the US Department of Agriculture under award number 2014-38502-22598 and USDA-NIFA. We thank Nathan Braun, Christopher Owusu, and Reshma Thapa for their help in the fieldwork; Agrisoma USA for supplying carinata seed; and the South Dakota Agricultural Experiment Station for support.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of the present study are available from the corresponding author upon reasonable request.

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How to cite this article: Bhattarai D, Abagandura GO, Nleya T, Kumar S. Responses of soil surface greenhouse gas emissions to nitrogen and sulfur fertilizer rates to *Brassica carinata* grown as a bio-jet fuel. *GCB Bioenergy*. 2021;13:627–639. https://doi.org/10.1111/gcbb.12784