

## ORIGINAL ARTICLE

## Soil Fertility and Crop Nutrition

# Sulfur fertilization sources for multisite sugarcane (*Saccharum* spp.) fields in Brazil

Rafael Otto<sup>1</sup>  | Lucas Miguel Altarugio<sup>1</sup> | Victor Xavier Rizzo<sup>1</sup> | Estêvão Vicari Mellis<sup>2</sup> | Sarah Tenelli<sup>1</sup> | Johnny Rodrigues Soares<sup>1</sup> | Sarah Mello Leite Moretti<sup>1</sup>

<sup>1</sup>Department of Soil Science, Luiz de Queiroz College of Agriculture, University of São Paulo (ESALQ/USP), Piracicaba, São Paulo, Brazil

<sup>2</sup>Soils and Environmental Resources Center, Agronomic Institute of Campinas (IAC), Campinas, São Paulo, Brazil

## Correspondence

Rafael Otto, Department of Soil Science, Luiz de Queiroz College of Agriculture, University of São Paulo (ESALQ/USP), Pádua Dias Avenue, 11, Piracicaba, São Paulo 13418-900, Brazil.  
Email: [rotto@usp.br](mailto:rotto@usp.br)

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## Abstract

Sulfur (S) deficiency in soils has become a common problem in Brazilian sugarcane (*Saccharum* spp.) fields due to high nutrient extraction by the crop without adequate replacement. Five field trials were conducted in Brazil over two consecutive ratoon cycles to evaluate S fertilizer sources and application rates and their residual effects on soil S availability and sugarcane yield. The treatments included (1) two S application rates (50 and 100 kg S ha<sup>-1</sup>), (2) four S sources (gypsum, elemental sulfur [ES], ammonium sulfate [AS], and polysulfate [PS]), and (3) a control (no S application). The PS and AS were applied in band over sugarcane rows, whereas gypsum and ES were broadcast during the first ratoon, subsequently, no S was reapplied and residual effects were assessed in the second ratoon. The broadcast application of gypsum at 100 kg S ha<sup>-1</sup> increased the available soil S concentration after 6 and 12 months. However, S levels remained below 10 mg dm<sup>-3</sup> after two ratoon harvests, regardless of the S source. Leaf S concentration increased after band application of PS at 100 kg S ha<sup>-1</sup> in both ratoon cycles and after broadcast application of ES and gypsum (residual effect) in the second ratoon. Stalk yield increased by an average of 5–7 Mg ha<sup>-1</sup> across sites in the first ratoon with S sources application. In the second ratoon, significant residual effects of S sources led to an increase in stalk yield, averaging 7–9 Mg ha<sup>-1</sup> compared to the control. Our findings indicate that S fertilization should be applied annually, with sulfate-based fertilizers preferred in the short term. ES shows potential for long-term S supply.

## Plain Language Summary

There is limited information on how different sulfur fertilizer sources, such as sulfate, polysulfate, and elemental sulfur, affect sugarcane yield and soil sulfur levels under different soil types and climatic conditions. This study evaluated both the immediate

**Abbreviations:** AS, ammonium sulfate; ES, elemental sulfur; PS, polysulfate; TRS, theoretical recoverable sugar.

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and residual effects of applying different rates and sources of sulfur fertilizers on soil sulfur availability and sugarcane production across two ratoon cycles at multiple sites in Brazil. Results showed that sulfur levels in the topsoil were below  $10 \text{ mg dm}^{-3}$  at most sampling points after sulfur application, highlighting the need for annual sulfur fertilization in sugarcane ratoon cycles. On average, sulfur application increased yields by  $5\text{--}7 \text{ Mg ha}^{-1}$  in the first ratoon and  $7\text{--}9 \text{ Mg ha}^{-1}$  in the second ratoon cycle (residual effects) compared to the control. Sulfate-based fertilizers effectively supplied sulfur in the short term, while elemental sulfur was better suited for long-term use, particularly in areas with straw coverage and naturally low sulfur levels in the soil.

## 1 | INTRODUCTION

Brazil is the leading producer of sugarcane (*Saccharum* spp.), accounting for 40% of global production. In Brazil, sugarcane cultivation occupies 8.4 million ha and produces 38.7 million t of sugar and 27.5 billion liters of ethanol annually (CONAB, 2023; FAO, 2021). Sulfur (S) is essential for proper sugarcane development and high yields. For a yield basis of  $100 \text{ Mg ha}^{-1}$  (fresh stalks), modern sugarcane varieties grown in Brazil allocate  $23 \text{ kg S } 100 \text{ t}^{-1}$  in the stalks and  $13 \text{ kg S } 100 \text{ t}^{-1}$  in dry leaves and green tops (Otto et al., 2019). In southeastern Brazil, where the majority of sugarcane is produced, the soil is mostly weathered Oxisols characterized by low nutrient availability, high acidity ( $\text{pH} < 5.0$ ), and low organic matter content (around 1%). These conditions, coupled with the high C:S ratios (around 500) of plant residues and sulfate leaching to deeper soil layers, can lead to S deficiency and be aggravated by the application of limestone and phosphate fertilizers (Pias et al., 2018).

Sulfur is an essential element for plant metabolism and biogeochemical cycles involving various oxidation and reduction reactions. Sulfur is present in the catalytic sites of enzymes and coenzymes, such as urease and coenzyme A, and participates in electron transport through iron (Fe) and S groups (Epstein & Bloom, 2005). This nutrient is also a component of four essential amino acids, proteins, coenzymes, and polysaccharide esters in plant organs, and participates in chlorophyll synthesis and non-photosynthetic fixation of atmospheric  $\text{CO}_2$  (Coleman, 1966; Crawford et al., 2000; Malavolta et al., 1997; Marschner, 1995). Soil is an important S reservoir, where a large proportion of total soil S is found in the organic fraction, of which 30%–75% are esters (Moreira & Siqueira, 2006). However, the organic S fraction must be mineralized by bacteria and fungi before being absorbed by plants. Soil solution sulfate ( $\text{SO}_4^{2-}$ ) is the major source of S taken up by crop roots (Marschner, 1995).

The mineralization and immobilization of S are controlled by the C:S ratio of the plant material. Soil  $\text{SO}_4^{2-}$ -S increases when the C:S ratio is less than 200 but is immobilized by

the soil microbiota when the C:S ratio is greater than 400 (Moreira & Siqueira, 2006). The availability of inorganic S ( $\text{SO}_4^{2-}$ ) is influenced by soil type. Inorganic S is adsorbed in clay-textured soils containing low-activity 1:1 clays, Fe or aluminum (Al) oxides, or hydroxides with pH-dependent ionizable OH groups (Venegas et al., 2007). In tropical soils with variable charge (clay fraction dominated by kaolinite and Fe and Al oxides),  $\text{SO}_4^{2-}$ -S adsorption may decrease with increasing soil pH. Thus, studies of S application in different soil types are critical to understanding  $\text{SO}_4^{2-}$  dynamics and its effects on soil and plant nutrition. In addition, applying lime can increase plant-available S in acidic soils, but may also increase  $\text{SO}_4^{2-}$ -S mobility and the risk of leaching to subsoil (Couto et al., 1979). The leaching for deeper soil layers may reduce plant uptake of S in the early stages of plant development. High rates of phosphate fertilization can also stimulate S leaching due to the competition of phosphate ions with  $\text{SO}_4^{2-}$ -S for adsorption sites (Pozza et al., 2007; Venegas et al., 2007).

Phosphogypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) (gypsum hereinafter), a byproduct of the phosphate industry, is commonly used as a soil amendment and S source (15% of  $\text{SO}_4^{2-}$ -S) in Brazil due to its high availability and relatively low cost (Caires & Guimarães, 2018). In sugarcane fields, gypsum ( $750\text{--}1000 \text{ kg ha}^{-1}$ , or about  $113\text{--}150 \text{ kg S ha}^{-1}$ ) is often applied biennially to improve subsoil conditions for root growth and provide S nutrition. However, in the second year, soil analysis usually reveals low S levels ( $<5 \text{ mg dm}^{-3}$ ) at 0- to 25-cm depth due to the high mobility of  $\text{SO}_4^{2-}$  in the soil profile, and the plant may be S deficient as most of the root systems occupy the top 50 cm. In these situations, applying other sulfate-based fertilizers, such as ammonium sulfate (AS) or polysulfate (PS) (Bhatt et al., 2021; Herrera et al., 2022; Pavinato et al., 2020), may help provide adequate S to meet sugarcane demands. PS is a natural mineral that gradually releases multiple nutrients (potassium [K], S, magnesium [Mg], and calcium [Ca]). Compared to applying conventional fertilizers such as  $\text{K}_2\text{SO}_4$ , PS application reduces nutrient leaching and may increase crop yields and S uptake (Yermiyahu et al.,

2017). However, PS has not been evaluated as an S source for sugarcane.

Another option to supply S to crops is the application of elemental sulfur (ES). ES fertilizers contain high S concentrations and thus can be added to fertilizer formulations without greatly reducing nitrogen (N), phosphorus (P), or K content (Horowitz & Meurer, 2006). ES fertilizers are usually mixed with bentonite to improve dispersion in the soil, resulting in a concentrated S source with 90% S (Lucheta & Lambais, 2012). However, once applied to the soil, ES must be oxidized to  $\text{SO}_4^{2-}$ -S for plant uptake (Horowitz & Meurer, 2007). ES oxidation is affected by environmental conditions (e.g., temperature, humidity, aeration, and soil pH), biological factors (e.g., microbial diversity), fertilizer properties (e.g., particle size, soil dispersion, and formulation), and fertilizer application methods (Lucheta & Lambais, 2012). Several studies have shown ES application increases sugarcane yield (Nicho et al., 2022; Wiedenfeld, 2011) and has residual effects in the soil that provide continuous S availability for subsequent crops with minimal S leaching losses (Riley et al., 2002; Szulc et al., 2012). Malik et al. (2021) reported greater  $\text{SO}_4^{2-}$  oxidation when ES fertilization was combined with organic amendments, such as filter cake from sugarcane cultivation. However, other studies have found no effect of ES application on sugarcane yield (McCray et al., 2018). Moreover, most studies of factors affecting the oxidation of ES in soil have used quick active powdered ES rather than granular ES (Chien et al., 2016).

Sulfur fertilization can increase sugarcane stalk diameter, height, and yield by 5%–10% (Kumar et al., 2011; Nicho et al., 2022; Wiedenfeld, 2011). However, studies have reported no effect of S addition on soil properties, such as organic matter content (Bologna-Campbell et al., 2013; McCray et al., 2018). Understanding the dynamics of S sources in different soils is necessary to avoid sugarcane yield limitations due to S deficiency. There is limited information on the impact of different S sources, including sulfate, PS, and ES, on sugarcane yield and soil S content across different soil types and climatic conditions. This study aimed to assess the immediate and residual effects of applying different rates and sources of S on soil S availability and sugarcane production parameters during two sugarcane ratoon cycles at multiple locations in Brazil.

## 2 | MATERIAL AND METHODS

### 2.1 | Description of the experimental areas

The field experiments were established at five sites with distinct edaphoclimatic characteristics representing the conditions of the main areas of sugarcane cultivation in Brazil (Figure 1, Table 1). The experimental sites were homogeneous with flat relief, and the available S concentration in the

#### Core Ideas

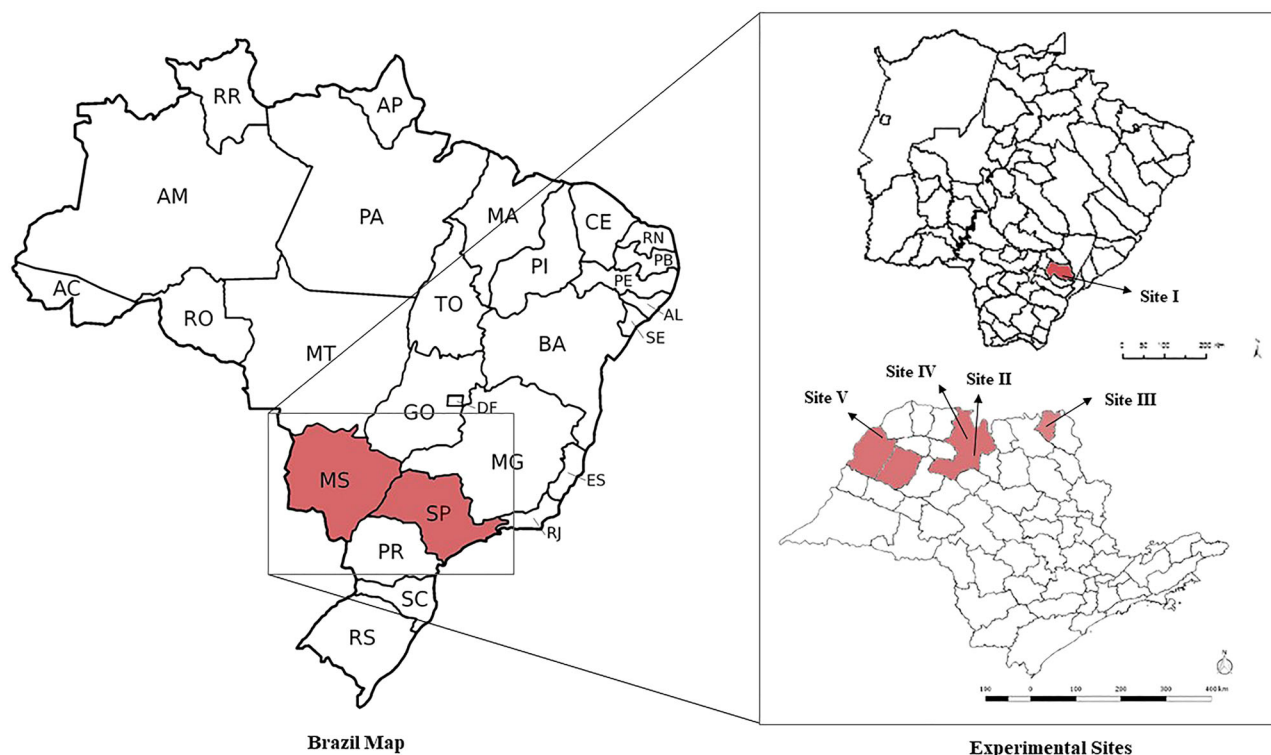
- Four S sources, two rates, and their residual effect were evaluated at five sites over two sugarcane cycles.
- Soil S concentration increased under gypsum at 100 kg ha<sup>-1</sup> in the first cycle, but it remained low afterward.
- On average of all sites, S sources increased stalk yield by 5–7 and 7–9 Mg ha<sup>-1</sup> in the first and second ratoons.
- Sulfate S fertilizers resulted in a positive effect on sugarcane yield in the short term.
- Elemental sulfur can be a promising S source in the long term for sugarcane ratoon production.

0–25 cm soil layer was less than 10 mg dm<sup>-3</sup>. The locations and soil types of the sites were as follows:

- (1) Site I, Ivinhema—Mato Grosso do Sul state (MS), (22° 17' 59.18" S; 53° 55' 7.49" W), Typic Kandiodox with sandy loam texture.
- (2) Site II, Cedral—São Paulo state (SP), (20° 58' 9.37" S; 49° 19' 55.52" W), Arenic Kandiodult with sandy loam texture.
- (3) Site III, Buritizal—São Paulo state (SP), (20° 16' 05.48" S; 47° 38' 40.66" W), Rhodic Hapludox with clay texture.
- (4) Site IV, Onda Verde—São Paulo state (SP), (20° 40' 47.00" S; 49° 10' 50.03" W), Typic Hapludox with sandy loam texture.
- (5) Site V, Suzanópolis—São Paulo state (SP), (20° 28' 49.59" S; 51° 6' 44.13" W), Typic Eutrudox with sandy loam texture (Soil Survey Staff, 2014).

In the sugarcane replanting period, before the cane plant planting (first sugarcane cycle), the following fertilization and management were performed at each site:

- (1) Site I: Application of lime (4.3 t ha<sup>-1</sup>) and gypsum (1.2 t ha<sup>-1</sup>);
- (2) Site II: Rotation with a leguminous crop (after five ratoon cycles) and application of vinasse, filter cake (sugarcane industrial byproducts), and lime (2.9 t ha<sup>-1</sup>);
- (3) Site III: Application of vinasse (applied annually only before the experiment establishment) and filter cake (20 t ha<sup>-1</sup>) was performed.
- (4) Site IV: Application of gypsum (0.9 t ha<sup>-1</sup>) and lime (1.4 t ha<sup>-1</sup>); and
- (5) Site V: Application of gypsum (0.5 t ha<sup>-1</sup>) and lime (1.5 t ha<sup>-1</sup>).



**FIGURE 1** Geographic locations of the experimental sites in south-central Brazil designed to assess the effects of sulfur application on sugarcane ratoon fields (site I: Ivinhema-MS; site II: Cedral-SP; site III: Buritizal-SP; site IV: Onda Verde-SP; and site V: Suzanópolis-SP).

In the sugarcane planting, all sites were fertilized according to Raij et al. (1997). The trials were established on the first ratoon after the first-year cane harvest (cane plant). Before the establishment of the experiments at each site, composite soil samples comprising 10 subsamples were collected from the 0 to 25, and 25 to 50 cm layers using an auger for chemical and physical characterization (Table 1). At all sites, sugarcane was planted under conventional tillage (soil disking or plowing) between February and June 2017. The cane plant (i.e., first harvest) was mechanically harvested between May and June 2018. The trials were then set up and conducted during two consecutive ratoon cycles harvested in 2019 and 2020 (Table S1). The water balance at each site is shown in Figure S1.

## 2.2 | Experimental design

A factorial  $2 \times 4 + 1$  experimental design was adopted with (1) two S application rates (50 and 100 kg S ha<sup>-1</sup>), (2) four S sources (gypsum, ES, AS, and PS), and (3) an additional treatment without S application (control), arranged in randomized blocks with four replications. The plots consisted of six 15-m-long sugarcane rows spaced 1.5 m apart. All the treatments received equal rates of 120 kg N ha<sup>-1</sup>, 50 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup>, and 150 kg K<sub>2</sub>O ha<sup>-1</sup>, as the treatment AS had N and PS had K in their composition, the ammonium nitrate and

potassium chloride were applied to reach the same N and K amount in all treatments. The fertilizers used were ammonium nitrate, triple superphosphate, and potassium chloride as standard ratoon fertilization after the first-year plant harvest in 2018. The standard ratoon fertilizer was applied in bands of 30 cm on each side over the sugarcane row without mechanical incorporation.

The experimental treatments for PS and AS were applied in bands over the sugarcane row at the soil surface, whereas gypsum and ES were applied in the total area (broadcast) over the soil surface, which is the common application method for these treatments. The gypsum used in the study was phosphogypsum, a byproduct of the P fertilizer industry, containing 19% Ca and 15% S as sulfate (adding 63 and 127 kg ha<sup>-1</sup> of Ca at the rates of 50 and 100 kg S ha<sup>-1</sup>, respectively). This product is often used as a soil conditioner and source of S for agricultural crops in Brazil. The ES source was a pellet with 3 mm of ES (“S<sup>0</sup>”) combined with 10% bentonite and contained 90% S (commercial product Sulfurgran, ICL). The PS (commercial product Polysulphate, ICL) was a natural crystal mineral (polyhalite) with a gradual release of nutrients, containing 14% K, 12% Ca, 3.6% Mg, and 19.2% S (adding 36.5, 31.3, and 9.4 kg ha<sup>-1</sup> of K, Ca, and Mg at the rate of 50 kg S ha<sup>-1</sup>, and 72, 62.5, and 18.8 kg ha<sup>-1</sup> of K, Ca, and Mg at the rate of 100 kg S ha<sup>-1</sup>). The AS contained 20% N as ammonium and 22% S as sulfate (commercial product AS, Fertipar).

TABLE 1 Soil chemical and physical characterization at five sugarcane fields (I–V) in south-central Brazil before sulfur experiment implementation in 2018.

| Sites    | pH  | CaCl <sub>2</sub> | C (g kg <sup>-1</sup> ) | P (mg dm <sup>-3</sup> ) | S (mg dm <sup>-3</sup> ) | K (mmol <sub>c</sub> dm <sup>-3</sup> ) | Ca (mmol <sub>c</sub> dm <sup>-3</sup> ) | Mg (mmol <sub>c</sub> dm <sup>-3</sup> ) | BS (%) | Sand (g kg <sup>-1</sup> ) | Clay (g kg <sup>-1</sup> ) | Silt (g kg <sup>-1</sup> ) |
|----------|-----|-------------------|-------------------------|--------------------------|--------------------------|-----------------------------------------|------------------------------------------|------------------------------------------|--------|----------------------------|----------------------------|----------------------------|
| 0–25 cm  |     |                   |                         |                          |                          |                                         |                                          |                                          |        |                            |                            |                            |
| I        | 5.1 | 0.6               | 12                      | 9                        | <0.9                     | 34                                      | 3.0                                      | 3.0                                      | 77     | 766                        | 187                        | 47                         |
| II       | 4.8 | 0.6               | 7                       | 5                        | 1.5                      | 4.0                                     | 4.0                                      | 4.0                                      | 44     | 796                        | 150                        | 54                         |
| III      | 4.7 | 1.6               | 8                       | 5                        | 3.2                      | 10                                      | 7.0                                      | 7.0                                      | 56     | 517                        | 380                        | 103                        |
| IV       | 5.9 | 0.6               | 9                       | 9                        | 0.9                      | 53                                      | 28                                       | 28                                       | 87     | 720                        | 250                        | 29                         |
| V        | 5.0 | 0.8               | 11                      | 6                        | 1.0                      | 29                                      | 5                                        | 5                                        | 78     | 808                        | 126                        | 65                         |
| 25–50 cm |     |                   |                         |                          |                          |                                         |                                          |                                          |        |                            |                            |                            |
| I        | 5.3 | 0.5               | 14                      | 12                       | <0.9                     | 28                                      | 2.0                                      | 2.0                                      | 73     | 788                        | 174                        | 38                         |
| II       | 4.5 | 0.5               | 6                       | 29                       | 1.0                      | 3.0                                     | 4.0                                      | 4.0                                      | 40     | 705                        | 225                        | 70                         |
| III      | 4.9 | 1.2               | 6                       | 8                        | 3.1                      | 12                                      | 5.0                                      | 5.0                                      | 61     | 495                        | 480                        | 26                         |
| IV       | 5.8 | 0.5               | 5                       | 28                       | <0.9                     | 12                                      | 5                                        | 5                                        | 59     | 687                        | 273                        | 39                         |
| V        | 5.4 | 0.3               | 8                       | 8                        | 0.9                      | 25                                      | 4                                        | 4                                        | 75     | 734                        | 177                        | 89                         |

Note: C, carbon content determined by the Walkley–Black method; the contents of P, K, Ca, and Mg were extracted using an ion exchange resin method (1 M NaHCO<sub>3</sub>, pH 8.5); S (0.01 M calcium phosphate); pH in CaCl<sub>2</sub> (0.01 M); and BS, bases saturation. Soil chemical analysis was performed according to Raij et al. (2001), and soil particle-size analysis was conducted as described by Blake and Hartge (1986). Site I, Ivinhema-MS; Site II, Cedral-SP; Site III, Buritizal-SP; Site IV, Onda Verde-SP; and Site V, Suzanópolis-SP.

After the first ratoon harvest in 2019 (Table 1), the standard ratoon fertilization with N, P<sub>2</sub>O<sub>5</sub>, and K<sub>2</sub>O applied in the first ratoon cycle was also repeated in all plots in the second ratoon cycle. Sulfur sources were not applied after the first ratoon harvest, and the residual effect of S application was evaluated in the second ratoon cycle.

## 2.3 | Measurements

At 6, 12, and 24 months after treatment application, soil sampling was conducted at 0- to 25-cm and 25- to 50-cm depths at sites I–IV to quantify available S concentration. Using a 5-cm-wide auger, six composite soil samples were collected from each plot at 25 cm from the sugarcane row, following the common practice of sugarcane growers. Sulfate (SO<sub>4</sub><sup>-2</sup>) was extracted by shaking the soil samples with 0.5 M calcium phosphate and quantified by turbidimetry according to Raij et al. (2001).

Leaf sampling was performed at sites I–IV in the summer during the phenological stage of initial stalk elongation. Fifteen top visible dewlap leaves were collected from each plot, and only the central parts of the leaves (excluding the midrib) were sent to the laboratory. The leaf samples were dried, milled, and digested with nitric acid, and the S concentration was determined by measuring the absorbance at 420 nm in a spectrophotometer (Malavolta et al., 1997).

At harvest, tiller number, stalk diameter, and height were measured in the four central rows of each plot. Tillers were counted in 15 m of each sugarcane row at sites I–V. Undeveloped tillers, that is, very thin or shorter than 0.5 m, were excluded. Stalk diameter and height were measured in 10 stalks per plot at sites II–IV in the first ratoon harvest and at sites I–IV in the second ratoon harvest. The stalk diameter was measured in the basal region of the stalk. Stalk height was measured from the base of the plant to the point of separation between the stalk and the green top.

Sugarcane yield was evaluated by mechanically harvesting the four central rows of each plot and weighing the stalk mass using an automated truck equipped with a load cell system at sites I–V for the first ratoon and sites I–IV for the second ratoon. Yield was not evaluated for the second ratoon at site V due to operational issues. Sugarcane yield was extrapolated to Mg ha<sup>-1</sup> on a fresh biomass basis. Prior to harvest, ten sugarcane stalks were collected from the central rows of each plot. The top and dry leaves were removed, and the stalks were sent to the laboratory of each sugarcane mill for analysis of industrial quality parameters (sucrose, fiber, and purity) according to Fernandes (2011). Theoretical recoverable sugar (TRS) (kg Mg<sup>-1</sup>) and sugar yield (Mg ha<sup>-1</sup>) were calculated considering the content of soluble solids (brix), apparent sucrose content, fiber, and purity, according to Fernandes (2011).

## 2.4 | Data analysis

Data normality was confirmed by the Shapiro–Wilk test ( $p > 0.10$ ), and no data transformations were required to meet the assumptions of analysis of variance (ANOVA). Data, excluding the control plots, were subjected to two-way ANOVA of interactions to test the effects of the S application rate (50 or 100 kg S ha<sup>-1</sup>) and S source on the soil S concentration, leaf S content, sugarcane production, and quality parameters at each site and ratoon cycle. One-way ANOVA was performed to compare the treatments containing S with the control treatment. When statistically significant ( $p \leq 0.10$ ), average values were compared using the least significant difference (LSD) test. All statistical analyses were performed using R software (R Development Core Team, 2020).

## 3 | RESULTS

### 3.1 | Soil S concentration

At site II, the available soil S concentration in the surface layer was 36% higher in the gypsum treatment compared to the other S treatments, 12 months after S application (Figure 2). At site III, 6 months after application, the available soil S concentration in the gypsum treatment was 83% higher in the 0–25 cm and 55% higher in the 25–50 cm layers, compared with the other S treatments. The S concentration with gypsum was 49% higher in the 25–50 cm layer after 12 months of application, and 3% higher in the 25–50 cm after 24 months of application, compared to other treatments at site III (Figure 2). In terms of S rate effects, the average across all sites showed that applying 100 kg S ha<sup>-1</sup> increased the available soil S concentration in the 25–50 cm layer by 9% and 59% compared to the 50 kg S ha<sup>-1</sup> rate and the control, respectively, 6 months after application (Table S2). After 24 months, the soil S concentration in the 0–25 cm layer was below 10 mg dm<sup>-3</sup> at all sites for all four S treatments and both S rates (Figure 2, Table S2).

### 3.2 | Leaf S concentration

In general, the S sources and/or rates increased leaf S concentration in sugarcane across all sites and ratoon cycles ( $p < 0.10$ ), except site III in the first ratoon and site II in second ratoon (Table 2). At site I, the sugarcane leaf S concentration in the first ratoon was higher after the broadcast application of gypsum at 100 kg S ha<sup>-1</sup> rate than gypsum broadcast at 50 kg S ha<sup>-1</sup> (Table 2). In the second ratoon, the residual effect of ES broadcast application at 100 kg S ha<sup>-1</sup> rate showed greater leaf S concentration levels compared with the residual effect of ES applied at 50 kg S ha<sup>-1</sup>. In the first

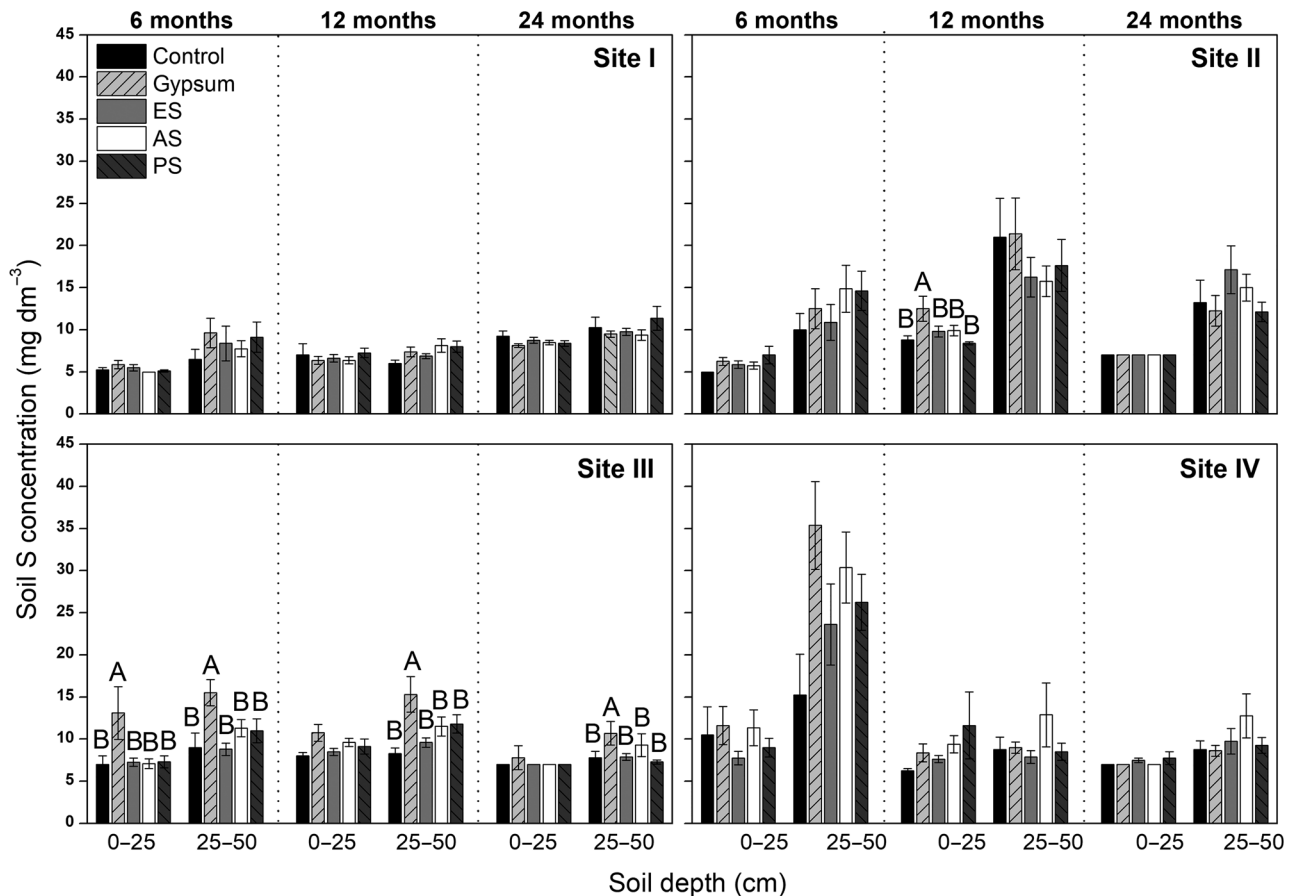
ratoon at site II, the band application of PS at 100 kg S ha<sup>-1</sup> resulted in a higher leaf S concentration than the application of other S sources. The broadcast application of ES at 50 kg S ha<sup>-1</sup> exhibited greater leaf S concentrations compared with the broadcast application of ES at 100 kg S ha<sup>-1</sup>. In the second ratoon at site III, the leaf S concentration was higher when PS or AS was band applied at 100 kg S ha<sup>-1</sup> than when gypsum was broadcast at 100 kg S ha<sup>-1</sup>. However, the leaf S concentration did not differ between the band application of AS and the broadcast application of ES at the same rate. In the second ratoon at site IV, the analysis of the interaction between S source and rate showed that the residual effects of gypsum or ES broadcast at 50 kg S ha<sup>-1</sup> revealed higher leaf S concentration compared to the residual effects of gypsum or ES broadcast at 100 kg S ha<sup>-1</sup> or band application of AS or PS at 50 kg S ha<sup>-1</sup>.

### 3.3 | Sugarcane production parameters

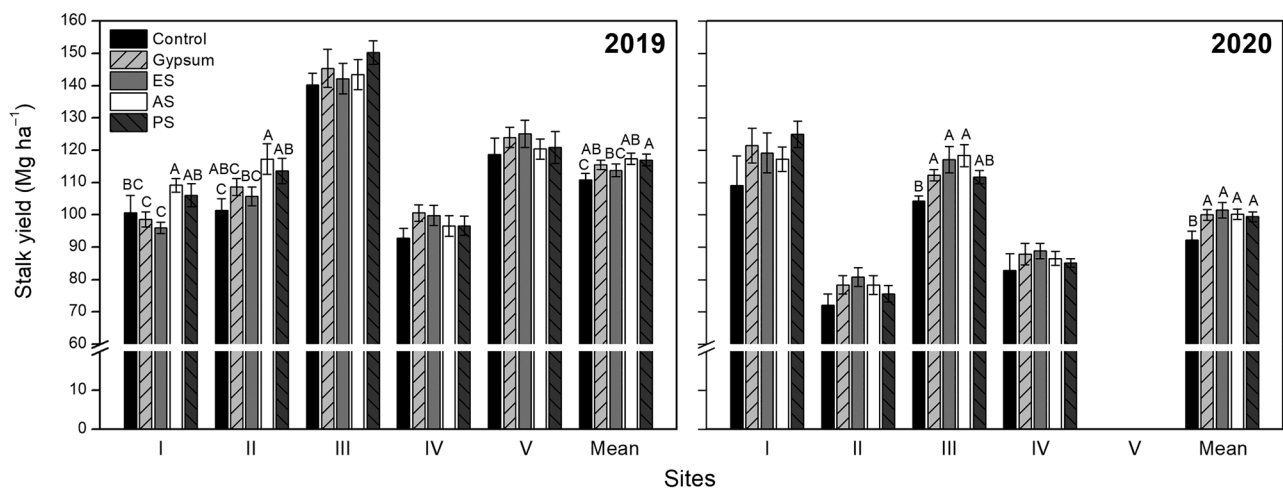
Sulfur fertilizer application did not affect the average number of sugarcane tillers or stalk diameter at the sites (Table S3). Sugarcane stalk height at the first ratoon harvest was influenced by the S rate and source. Compared with the control, stalk height was significantly greater when S sources were applied at 100 kg S ha<sup>-1</sup> and was greatest when AS was band applied ( $p < 0.10$ ).

Considering the average of both S rates at the first ratoon harvest, a positive effect of S fertilization on stalk yield was observed at sites I and II (Figure 3). At site I, band application of AS showed higher stalk yield in the first ratoon cycle by 8.5 Mg ha<sup>-1</sup> compared to the control and 11.9 Mg ha<sup>-1</sup> compared to the broadcast application of ES or gypsum. The stalk yield under band application of PS was similar to those under band application of AS and the control. At site II, band application of AS or PS exhibited greater stalk yield by 15.9 and 12.2 Mg ha<sup>-1</sup>, respectively, versus the control. The stalk yield in the PS treatment was similar to those in the treatments in which gypsum or ES was broadcast, and the stalk yields in the latter treatments were similar to those in the control.

In the second ratoon cycle, sugarcane did not respond to the residual effects of S fertilization at sites I and II (Figure 3). At site III, the residual effects of gypsum, ES, and AS application on stalk yield were similar, with an average stalk yield gain of 11.6 Mg ha<sup>-1</sup> compared to the control. The stalk yield in the PS treatment at site III was similar to the control. When averaged across the five sites, S application (except ES) increased stalk yield by 5–7 Mg ha<sup>-1</sup> in the first ratoon cycle and by 7–9 Mg ha<sup>-1</sup> (all S sources) in the second ratoon cycle versus the control (Figure 3, Table S4). Overall, there were no differences in sugarcane yield between the 50 and 100 kg S ha<sup>-1</sup> rates, nor between S rates with the control across the sites (Table S4).



**FIGURE 2** Available soil S concentration at 6, 12, and 24 months (2018, 2019, and 2020) after S fertilizer application to sugarcane ratoons at sites I, II, III, and IV in south-central Brazil. The results for the S treatments are the average of the S rates (50 and 100 kg S ha<sup>-1</sup>). Different uppercase letters indicate significant differences among S fertilizer sources at the same soil depth according to the least significant difference (LSD) test ( $p < 0.10$ ). AS, ammonium sulfate; ES, elemental sulfur; PS, polysulfate.



**FIGURE 3** Sugarcane stalk yield at sites I–V and mean yield across sites in the first (2019) and second (2020) ratoon harvests after application of S fertilizers in south-central Brazil. Different uppercase letters indicate significant differences among S sources at the same site, according to the least significant difference (LSD) test ( $p < 0.10$ ). In the second ratoon harvest, S fertilizer was not applied, and the residual effect of S fertilization was evaluated. Site V was not evaluated in the second ratoon harvest due to operational issues. AS, ammonium sulfate; ES, elemental sulfur; PS, polysulfate.

**TABLE 2** Leaf S concentration in the first and second sugarcane ratoon cycles after the application of different S rates and sources at four sites (I–IV) in south-central Brazil.

| Treatments                                 | S rate (kg S ha <sup>-1</sup> ) | First ratoon cycle (2019) |        |      |       | Second ratoon cycle (2020) |      |        |        |
|--------------------------------------------|---------------------------------|---------------------------|--------|------|-------|----------------------------|------|--------|--------|
|                                            |                                 | I                         | II     | III  | IV    | I                          | II   | III    | IV     |
| Leaf S concentration (g kg <sup>-1</sup> ) |                                 |                           |        |      |       |                            |      |        |        |
| Gypsum                                     | 50                              | 1.20b                     | 2.53   | 1.53 | 1.83  | 1.13                       | 1.53 | 2.85   | 1.38Aa |
| ES                                         | 50                              | 1.30                      | 2.43a  | 1.60 | 1.28  | 1.08b                      | 1.57 | 3.06   | 1.39Aa |
| AS                                         | 50                              | 1.65                      | 2.55   | 1.73 | 1.25  | 1.06                       | 1.42 | 2.68   | 1.02B  |
| PS                                         | 50                              | 1.60                      | 2.78b  | 1.48 | 1.68  | 1.16                       | 1.47 | 2.85   | 1.01B  |
| Gypsum                                     | 100                             | 1.83a                     | 2.63B  | 1.68 | 2.05  | 1.24                       | 1.56 | 2.57C  | 0.97b  |
| ES                                         | 100                             | 1.58                      | 1.63Cb | 1.58 | 2.00  | 1.23a                      | 1.46 | 2.83BC | 1.02b  |
| AS                                         | 100                             | 1.15                      | 2.35B  | 1.80 | 1.90  | 1.13                       | 1.41 | 3.05AB | 0.94   |
| PS                                         | 100                             | 1.60                      | 3.50Aa | 1.93 | 1.73  | 1.17                       | 1.60 | 3.31A  | 0.98   |
| Control                                    |                                 | 1.73                      | 2.13b  | 1.30 | 1.28c | 1.05b                      | 1.45 | 2.65   | 1.40a  |
| Rate 50                                    |                                 | 1.44                      | 2.57a  | 1.58 | 1.51b | 1.11ab                     | 1.50 | 2.86   | 1.19b  |
| Rate 100                                   |                                 | 1.54                      | 2.53a  | 1.74 | 1.92a | 1.19a                      | 1.50 | 2.94   | 0.98c  |
| Control                                    |                                 | 1.73                      | 2.13BC | 1.30 | 1.28  | 1.05                       | 1.45 | 2.65   | 1.40A  |
| Gypsum                                     |                                 | 1.51                      | 2.58B  | 1.60 | 1.94  | 1.18                       | 1.54 | 2.71   | 1.17B  |
| ES                                         |                                 | 1.44                      | 2.03C  | 1.59 | 1.64  | 1.15                       | 1.51 | 2.95   | 1.20B  |
| AS                                         |                                 | 1.40                      | 2.45AB | 1.76 | 1.58  | 1.09                       | 1.41 | 2.86   | 0.97C  |
| PS                                         |                                 | 1.60                      | 3.14A  | 1.70 | 1.70  | 1.17                       | 1.53 | 3.08   | 0.99C  |
| p S source                                 |                                 | ns                        | 0.001  | ns   | ns    | ns                         | ns   | ns     | 0.003  |
| p S rate                                   |                                 | ns                        | ns     | ns   | 0.001 | 0.027                      | ns   | ns     | 0.001  |
| p S source × S rate                        |                                 | 0.013                     | 0.020  | ns   | ns    | 0.101                      | ns   | 0.038  | 0.005  |

Note: Different lowercase letters in the same column indicate significant differences among S rates (control, rate 50, and rate 100) or between 50 and 100 kg S ha<sup>-1</sup> rates for the same S source (gypsum, ES, AS, or PS), while different uppercase letters in the same column indicate significant differences among S sources only (control, gypsum, ES, AS, or PS) or among S sources for the same S rate (50 or 100 kg S ha<sup>-1</sup>) according to the LSD test ( $p < 0.10$ ). ns, no significant difference was found at the 10% level of significance.

Abbreviations: AS, ammonium sulfate; ES, elemental sulfur; LSD, least significant difference; PS, polysulfate.

### 3.4 | Theoretical recoverable sugar and sugar yield

In the first ratoon cycle, S application influenced TRS at site IV (Table 3). The TRS was similar in the PS, ES, and control treatments and lowest in the gypsum treatments. The TRS was 4% higher in the AS treatment than in the control ( $p < 0.10$ ). In the second ratoon cycle, the residual effects of all S sources increased TRS at site II compared to the control ( $p < 0.10$ ).

The sugar yield (Mg ha<sup>-1</sup>) differed among S sources at sites I and II in the first ratoon cycle (Table 4). At site I, sugar yield was 16% higher when AS was band applied versus other S sources at 50 kg S ha<sup>-1</sup>. At 100 kg S ha<sup>-1</sup> rate at this site, sugar yield was higher when PS or AS was band applied than when ES was broadcast but was similar when AS was band applied or gypsum was broadcast. At site II, applying AS and PS showed greater sugar yield compared with applying ES or the control, and gypsum treatment was similar to those in the other treatments. In the second ratoon cycle at site III, the residual effects of AS and ES exhibited greater sugar yield

compared with the control, whereas sugar yield in the PS and gypsum treatments was similar to the control.

## 4 | DISCUSSION

### 4.1 | Gypsum application improved soil S availability during sugarcane ratoon cycles

In Brazilian sugarcane fields, S deficiency has a significant impact on agriculture, mainly due to the critically lower levels of S content of tropical-weathered soils (Pias et al., 2018). The situation is aggravated by the frequent use of low-S NPK fertilizers, and the application of gypsum only every 2–7 years during ratoon cycles (Pias et al., 2018; Spironello et al., 1997), which further reduce S inputs. In this study, all five sites had surface soil S concentration below 10 mg dm<sup>-3</sup> (Figure 2), requiring S fertilization (Raij et al., 1997).

Sulfate-based fertilizers, which have greater solubility than ES, likely increased the concentration of available SO<sub>4</sub><sup>2-</sup> in the soil solution more rapidly for sugarcane uptake (Figure 2).

**TABLE 3** Theoretical recoverable sugar (TRS) in sugarcane stalk ratoon harvests after the application of different S rates and sources at sites I–V (first ratoon) and sites I–IV (second ratoon) in south-central Brazil.

| Treatments                                           | S rates (kg S ha <sup>-1</sup> ) | First ratoon cycle (2019) |       |       |          |       | Second ratoon cycle (2020) |        |       |       |
|------------------------------------------------------|----------------------------------|---------------------------|-------|-------|----------|-------|----------------------------|--------|-------|-------|
|                                                      |                                  | I                         | II    | III   | IV       | V     | I                          | II     | III   | IV    |
| Theoretical recoverable sugar (kg Mg <sup>-1</sup> ) |                                  |                           |       |       |          |       |                            |        |       |       |
| Gypsum                                               | 50                               | 160.4                     | 151.8 | 162.0 | 153.8    | 159.4 | 164.0                      | 135.4  | 171.2 | 147.4 |
| ES                                                   | 50                               | 160.0                     | 151.0 | 161.9 | 160.1    | 151.2 | 156.2                      | 126.2  | 175.2 | 147.9 |
| AS                                                   | 50                               | 161.4                     | 156.2 | 166.4 | 164.4    | 161.6 | 159.5                      | 127.1  | 178.6 | 148.9 |
| PS                                                   | 50                               | 159.6                     | 158.6 | 161.9 | 162.6    | 158.2 | 158.4                      | 127.6  | 173.0 | 146.9 |
| Gypsum                                               | 100                              | 159.0                     | 158.9 | 164.1 | 160.5    | 156.3 | 159.4                      | 126.3  | 172.6 | 144.3 |
| ES                                                   | 100                              | 158.9                     | 153.4 | 167.4 | 163.0    | 155.6 | 162.1                      | 127.8  | 175.5 | 150.3 |
| AS                                                   | 100                              | 156.7                     | 155.0 | 161.0 | 164.8    | 157.2 | 157.0                      | 124.4  | 174.6 | 145.9 |
| PS                                                   | 100                              | 158.2                     | 158.3 | 162.2 | 161.9    | 162.5 | 159.2                      | 134.3  | 174.4 | 143.9 |
| Control                                              |                                  | 155.6                     | 157.4 | 164.4 | 158.4    | 157.5 | 160.6                      | 119.3  | 173.9 | 143.3 |
| Rate 50                                              |                                  | 160.3                     | 154.4 | 163.0 | 160.3    | 157.6 | 159.5                      | 129.1  | 174.5 | 147.8 |
| Rate 100                                             |                                  | 158.2                     | 156.4 | 163.7 | 162.5    | 157.9 | 159.4                      | 128.2  | 174.3 | 146.1 |
| Control                                              |                                  | 155.6                     | 157.4 | 164.4 | 158.4BC  | 157.5 | 160.6                      | 119.3B | 173.9 | 143.3 |
| Gypsum                                               |                                  | 159.7                     | 155.4 | 163.0 | 157.1C   | 157.8 | 161.7                      | 130.9A | 171.9 | 145.9 |
| ES                                                   |                                  | 159.4                     | 152.2 | 164.7 | 161.6ABC | 153.4 | 159.2                      | 127.0A | 175.4 | 149.1 |
| AS                                                   |                                  | 159.0                     | 155.6 | 163.7 | 164.6A   | 159.4 | 158.3                      | 125.7A | 176.6 | 147.4 |
| PS                                                   |                                  | 158.9                     | 158.5 | 162.0 | 162.2AB  | 160.3 | 158.8                      | 130.9A | 173.7 | 145.4 |
| p S source                                           |                                  | ns                        | ns    | ns    | 0.082    | ns    | ns                         | 0.084  | ns    | ns    |
| p S rate                                             |                                  | ns                        | ns    | ns    | ns       | ns    | ns                         | ns     | ns    | ns    |
| p S source × S rate                                  |                                  | ns                        | ns    | ns    | ns       | ns    | ns                         | ns     | ns    | ns    |

Note: Different uppercase letters in the same column indicate significant differences among S sources (control, gypsum, ES, AS, or PS) according to the LSD test ( $p < 0.10$ ). ns, no significant difference at the 10% level. In the second ratoon harvest, S fertilizer was not applied and the residual effect was evaluated. Site V was not evaluated in the second ratoon harvest due to operational issues.

Abbreviations: AS, ammonium sulfate; ES, elemental sulfur; LSD, least significant difference; PS, polysulfate.

The low soil S levels during the first ratoon cycle after broadcast application of ES may be related to the slow oxidation of ES in the soil. ES dust is explosive, therefore, it is combined with bentonite to reduce the risk of explosion, resulting in a fertilizer with high S concentration, slow-release properties, and minimal leaching losses (Lucheta & Lambais, 2012). ES becomes available to plants after being oxidized to  $\text{SO}_4^{2-}$  by soil microorganisms, whose activity is influenced by several factors such as soil organic matter content, higher temperature, clay content, moisture, fertilizer particle size, pH, and management practices (Horowitz & Meurer, 2007). The slow oxidation of ES granulated with bentonite likely reduced the initial availability of  $\text{SO}_4^{2-}$  in the soil during the first 6 months of the experiment (Figure 2). During the initial oxidation process, the soil around bentonite-coated ES granules starts to acidify (Lucheta & Lambais, 2012), potentially slowing the oxidation process by inhibiting solubilizing microorganisms (Degryse et al., 2016).

The oxidation of ES can decrease soil pH (Lucheta & Lambais, 2012). Wiedenfeld (2011) reported minimal effects of ES application on soil pH in the first sugarcane plant cycle, but in the second and third ratoon cycles, ES reduced the soil

pH from 8.0 to 6.0 at application rates as high as 1120 kg S ha<sup>-1</sup>. Sulfate-based fertilizers typically have little or no effect on soil pH, except for AS, which can decrease soil pH due to the nitrification of its  $\text{NH}_4^+$ -N content (Cantarella et al., 2007; Fageria et al., 2010). In the present study, soil pH was not evaluated after S application, but significant changes were unlikely given the relatively low S rates (50 and 100 kg ha<sup>-1</sup> for two cycles). Moreover, the broadcast application of ES probably minimized its effect on soil pH compared to the band application. On the other hand, long-term use of ES and AS may reduce soil pH, while PS and gypsum are not expected to do so. These effects on soil pH could influence sugarcane growth and warrant further investigation, especially in tropical acidic soils.

Broadcast application of gypsum was the most effective for increasing soil S availability, resulting in increases of 55%–83% after 6 months and 49%–33% after two ratoon harvests, compared to the other sources and control (Figure 2). These increases are likely due to the moderate solubility of gypsum (Borgmann et al., 2021) and the effectiveness of broadcast application. In contrast, other sulfate S sources (AS and PS) were band applied, and soil was sampled 25 cm from the

**TABLE 4** Sugar yield during ratoon cycles after the application of different S rates and sources at sites I–V (first ratoon) and sites I–IV (second ratoon) in south-central Brazil.

| Treatments                         | S rates (kg S ha <sup>-1</sup> ) | First ratoon cycle (2019) |        |      |      |      | Second ratoon cycle (2020) |      |        |      |
|------------------------------------|----------------------------------|---------------------------|--------|------|------|------|----------------------------|------|--------|------|
|                                    |                                  | I                         | II     | III  | IV   | V    | I                          | II   | III    | IV   |
| Sugar yield (Mg ha <sup>-1</sup> ) |                                  |                           |        |      |      |      |                            |      |        |      |
| Gypsum                             | 50                               | 15.6b                     | 16.9   | 22.7 | 14.9 | 19.3 | 19.3                       | 10.3 | 18.9   | 13.0 |
| ES                                 | 50                               | 15.5b                     | 15.6   | 23.0 | 15.5 | 18.9 | 19.1                       | 9.9  | 21.3   | 12.9 |
| AS                                 | 50                               | 18.3a                     | 17.4   | 23.5 | 15.8 | 19.1 | 19.0                       | 10.0 | 21.0   | 12.7 |
| PS                                 | 50                               | 16.1b                     | 18.1   | 23.9 | 15.9 | 19.1 | 19.5                       | 9.4  | 18.9   | 12.7 |
| Gypsum                             | 100                              | 15.8bc                    | 16.8   | 24.8 | 16.8 | 19.8 | 20.0                       | 10.2 | 19.7   | 12.7 |
| ES                                 | 100                              | 15.1c                     | 16.6   | 23.8 | 16.7 | 19.4 | 18.8                       | 10.7 | 19.7   | 13.5 |
| AS                                 | 100                              | 16.4ab                    | 19.1   | 23.5 | 16.0 | 19.3 | 18.1                       | 9.8  | 20.8   | 12.8 |
| PS                                 | 100                              | 17.5a                     | 17.9   | 24.8 | 15.4 | 19.6 | 20.2                       | 10.5 | 19.9   | 12.2 |
| Control                            |                                  | 15.7                      | 15.9   | 23.1 | 14.7 | 18.7 | 17.5                       | 8.6  | 18.1   | 11.9 |
| Rate 50                            |                                  | 16.4                      | 17.0   | 23.3 | 15.5 | 19.1 | 19.2                       | 9.9  | 20.0   | 12.8 |
| Rate 100                           |                                  | 16.2                      | 17.6   | 24.2 | 16.2 | 19.5 | 19.3                       | 10.3 | 20.0   | 12.8 |
| Control                            |                                  | 15.7                      | 15.9B  | 23.1 | 14.7 | 18.7 | 17.5                       | 8.6  | 18.1C  | 11.9 |
| Gypsum                             |                                  | 15.7                      | 16.9AB | 23.8 | 15.8 | 19.6 | 19.6                       | 10.2 | 19.3BC | 12.8 |
| ES                                 |                                  | 15.3                      | 16.1B  | 23.4 | 16.1 | 19.2 | 18.9                       | 10.3 | 20.5AB | 13.2 |
| AS                                 |                                  | 17.4                      | 18.2A  | 23.5 | 15.9 | 19.2 | 18.5                       | 9.9  | 20.9A  | 12.8 |
| PS                                 |                                  | 16.9                      | 18.0A  | 24.4 | 15.6 | 19.4 | 19.9                       | 9.9  | 19.4BC | 12.4 |
| p S source                         |                                  | ns                        | 0.028  | ns   | ns   | ns   | ns                         | ns   | 0.024  | ns   |
| p S rate                           |                                  | ns                        | ns     | ns   | ns   | ns   | ns                         | ns   | ns     | ns   |
| p S source × S rate                |                                  | 0.036                     | ns     | ns   | ns   | ns   | ns                         | ns   | ns     | ns   |

Note: Different uppercase letters in the same column indicate differences among S sources (control, gypsum, ES, AS, or PS), while different lowercase letters in the same column indicate differences among S sources (gypsum, ES, AS, or PS) for the same S rate (50 or 100 kg S ha<sup>-1</sup>) according to the LSD test ( $p < 0.10$ ). ns, no significant difference at the 10% level. In the second ratoon cycle, S fertilizer was not applied and the residual effect was evaluated. Site V was not evaluated in the second ratoon cycle due to operational issues.

Abbreviations: AS, ammonium sulfate; ES, elemental sulfur; LSD, least significant difference; PS, polysulfate.

crop row, which may have limited their effect on soil S levels. However, after the second ratoon harvest, the available S concentration in the 0–25 cm layer was below 10 mg dm<sup>-3</sup>, regardless of the S source or application rate (Table S2). Interestingly, an increasing trend in available S concentration was observed in the 25–50 cm layer at site II, possibly due to SO<sub>4</sub><sup>2-</sup> leaching. This possible leaching and subsequent reduction in soil S concentration at the surface may be attributed to the high rainfall during this period (Figure S1) and the temporary increase in soil pH caused by the lime application.

The correction of soil pH through lime application in tropical soils increases negative charges (OH<sup>-</sup>), which not only reduces SO<sub>4</sub><sup>2-</sup> adsorption and thus increases soil S availability but may also increase SO<sub>4</sub><sup>2-</sup> leaching to the subsurface (Venegas et al., 2007). The formation of a neutral ion pair with Ca<sup>2+</sup> can further enhance SO<sub>4</sub><sup>2-</sup> mobility along the soil profile (Antonangelo et al., 2017). The application of phosphate-based fertilizers may also contribute to SO<sub>4</sub><sup>2-</sup> movement within the soil due to competition with phosphate ions for adsorption sites (Pozza et al., 2007). Consequently,

S fertilization should be performed annually to sustain soil S levels and avoid potential leaching risks. Special attention should be given to sandy soils with low fertility and a predominance of macropores, where faster internal water drainage may favor S losses through leaching (Tiecher et al., 2013). In such soils, sugarcane straw retention can (1) provide a long-term source of S, (2) improve soil organic matter content and chemical and physical properties, for example, cation exchange and water retention, and (3) ultimately boost sugarcane growth and yield (Gmach et al., 2019; Tenelli et al., 2019). Additional management practices, including crop rotation and the application of vinasse and filter cake, can further support sugarcane's S nutritional needs and sustain yields in sandy soils (McNunn et al., 2020; Zhao et al., 2020).

## 4.2 | Sulfate-based fertilization increased leaf S concentrations in the first ratoon cycle

Sulfate-based fertilizers increased the S concentration in sugarcane leaves, as well as stalk height, yield, and quality. These

improvements may be the result of adequate S release to the crop, enhancing its metabolic and physiological processes. Sulfur is a key structural element in the amino acids methionine and cysteine, and it serves as a cofactor in the synthesis of proteins, enzymes, and chlorophyll. Thus, S plays an essential role in maintaining crop yield and quality (Tandon, 1986).

During the first ratoon cycle at site II, sulfate-based fertilizers, particularly PS, raised leaf S concentrations compared with ES, gypsum, and the control (Table 2). At this site, most treatments resulted in sugarcane leaf S concentrations within the critical range of 1.3–2.8 g S kg<sup>-1</sup> (Anderson & Bowen, 1990). However, the band application of PS at 100 kg S ha<sup>-1</sup> increased leaf S concentrations above this range, reaching levels greater than 3.5 g S kg<sup>-1</sup>. Similar effects were observed at site III in the second ratoon cycle (Table 2).

Polyhalite, a slow-release multi-nutrient fertilizer containing ~19% low-solubility S (Herrera et al., 2022), may have provided a gradual release of S that supported sugarcane nutrition and increased leaf S concentrations at some sites (Table 2). By contrast, other sulfate-based fertilizers, such as gypsum, had little effect on leaf S concentrations in the first ratoon cycle. The high mobility of gypsum may have led to an early S release and a subsequent reduction in soil S availability, possibly due to crop uptake and leaching at most sites (Figure 3). The broadcast application of ES showed higher leaf S concentrations during the second ratoon season at sites I and IV, suggesting a good residual effect of ES on sugarcane nutrition.

### 4.3 | Sugarcane ratoon was responsive to S fertilization and residual effects differed by S source

The application of S sources influenced sugarcane stalk yield in both ratoon cycles (Figure 3). Applying sulfate-based fertilizers in the crop row (band) increased sugarcane stalk yield in the first ratoon harvest by 8.5–15.9 Mg ha<sup>-1</sup>. Similar results have been reported in the literature, with Kumar et al. (2011) showing that S application increased sugarcane stalk yield by 5.2%–10.5% compared to the control. At two of the five sites evaluated, sugarcane stalk yield was significantly higher under sulfate-based fertilizers (AS and PS) than under gypsum, whereas ES had no effect on sugarcane stalk yield versus the control (Figure 3). The response to S fertilization in the first ratoon cycle may have been even greater if not for the severe drought that occurred in most of south-central Brazil in December and January (summer of 2019) (Figure S1).

The lack of an effect of ES on sugarcane stalk yield during the first ratoon probably reflects the limited S availability from ES, regardless of soil S concentrations at 25- to 50-cm depth, as noted in previous studies (Malavolta & Moraes,

2007; Prochnow et al., 2010). At sites I and II, the slow oxidation of ES and initially low soil S levels likely contributed to the weak sugarcane response to ES application during the first ratoon cycle. The effectiveness of ES oxidation depends on soil conditions, which may have played a critical role in the yield response. This pattern is similar to that observed in corn (*Zea mays*) fields by Szulc et al. (2012), where a positive effect of ES on grain yield was seen only after 4 years of ES application. Malik et al. (2021) also reported that organic amendments, especially filter cake from sugarcane cultivation, increased the oxidation of ES to SO<sub>4</sub><sup>2-</sup>. Soil C concentration was low at sites I and II, where ES had lower efficiency than other S sources during the first ratoon cycle. However, at site III, which had higher soil organic matter content than the other sites (Table 1), the residual effects of ES in the second ratoon cycle were similar to those of AS and gypsum. Future studies should investigate the impact of ES in soils with relatively high organic matter content or those receiving long-term organic amendments.

The lack of sugarcane yield response to ES application in the first ratoon cycle may also be related to the particle size of the material. Chien et al. (2009) reported that the oxidation rate of granular ES mixed with bentonite was lower than powdered ES in sandy soil, despite the disintegration and dispersion of the granular ES in the soil. They concluded that although the disintegrated ES particles are very fine, they remain concentrated in the application zone, which limits contact with soil microorganisms and slows oxidation. Similarly, Chien et al. (2016) found that the granular form of ES may not benefit crops planted immediately after its application.

In the second ratoon cycle at site III, the residual effects of gypsum, AS, and ES application were similar, significantly increasing sugarcane stalk yield by 11.6 Mg ha<sup>-1</sup> compared to the control (Figure 3). This suggests ES application provided medium-term benefits. Although sugarcane at site III did not respond to S fertilization in the first ratoon cycle, the high stalk yield (>140 Mg ha<sup>-1</sup>) may have increased S extraction, explaining the response to S sources in the second ratoon cycle. The microbiota at site III may have oxidized ES more efficiently due to the higher organic matter content (Table 1). In an isotope-tracing study in Brazil, Degryse et al. (2020) found higher S recovery by corn and soybean [*Glycine max* (L.) Merr.] under ES application than sulfate-based sources. They attributed this to the rapid oxidation of ES and high leaching by sulfate-based sources in this environment compared to colder climates. Another study reported that ES oxidation is initially slow, leading to low fertilizer efficiency. But in subsequent years, plant S recovery matches or exceeds that in soils treated with sulfate-based fertilizers, as ES continues to release available S and is less prone to leaching (Degryse et al., 2021). Consequently, ES can positively impact sugarcane yield and may have similar or greater efficiency than other S sources like gypsum (Nicchio

et al., 2022). Therefore, depending on the soil, ES could be an effective S source for sugarcane in the medium- or long-term, whereas sulfate-based fertilizers may be preferred for short-term nutrient supply.

Sulfur fertilization in sugarcane can have high profitability. For example, S sources increased stalk yield by 5–7 Mg ha<sup>-1</sup> in the first ratoon and 7–9 Mg ha<sup>-1</sup> in the second ratoon (residual effect), resulting in a total increase of 12–16 Mg ha<sup>-1</sup>. With a stalk price of \$23 Mg<sup>-1</sup>, harvest costs of \$6 Mg<sup>-1</sup> (UDOP, 2024), and gypsum priced at \$27 Mg<sup>-1</sup> (\$0.18 kg<sup>-1</sup> of S) (GlobalFert, 2024), the economic return from S fertilization would range from \$190 to \$258 ha<sup>-1</sup>.

#### 4.4 | Sulfate-based fertilizers improved sugarcane quality

An adequate S supply is necessary for sustainably enhancing sugarcane yield and quality. In the first ratoon cycle, AS and PS application showed higher TRS at site IV and sugar yield at sites I and II (Tables 3 and 4). These increases may be attributed to the role of S in carbohydrate metabolism (Malavolta, 1976). In contrast, ES application had little effect on TRS and sugar yield in the first ratoon cycle, probably because of lower soil S availability. However, in the second ratoon cycle, the residual effects of ES and AS application led to an increase in sugar yield at site III compared to the other S sources. This aligns with McCray et al. (2018), who also reported that S application boosts sugar yields.

#### 4.5 | Implications of S fertilization for the sugarcane ratoon system

Sulfur fertilization can increase the available soil S concentration directly through S fertilizer inputs and indirectly by increasing sugarcane yields, leading to more straw deposition and subsequent S release upon decomposition. In this study, the S contribution from fertilizers and sugarcane straw over two ratoon cycles was probably lower than the total S stored in soil organic matter, and thus insufficient to affect the C/S ratio and alter soil S concentration. Over the two ratoon cycles, S fertilization added 50 or 100 kg S ha<sup>-1</sup>, while sugarcane straw added ~40 kg S ha<sup>-1</sup> (20 kg S ha<sup>-1</sup> per ratoon) to the system. These inputs are small compared to the total S stored in the soil, where the organic S fraction must be mineralized by soil microbiota to become available to plants. However, soil S concentrations did not change at sites I and IV, remaining below 10 mg dm<sup>-3</sup> (Figure 2). The C/S ratio of soil organic matter probably remained higher than the C/S ratio provided by fertilizer and straw, due to their lower S input, resulting in only slight changes in soil S concentration.

Overall, applying S fertilizer at 100 kg S ha<sup>-1</sup> showed higher soil S availability after two ratoon cycles, but S levels in the 0–25 cm layer largely remained in the deficient range (<10 mg dm<sup>-3</sup>). These findings indicate that monitoring S concentration in the surface layer is essential for sustainable fertilizer management in sugarcane ratoon cycles. Additionally, the low soil S levels indicate annual S applications, rather than the current biennial recommendation, may be necessary to prevent SO<sub>4</sub><sup>2-</sup> leaching into deeper soil layers.

The band application of S as sulfate at the crop row position tended to improve sugarcane nutrition compared to broadcast gypsum or ES. Therefore, sulfate-based fertilizers should be preferred as short-term S sources in deficient soils. Broadcast application may limit plant root contact for SO<sub>4</sub><sup>2-</sup> uptake, resulting in lower efficiency of the S source, as observed for gypsum compared to band application of sulfate-S sources (AS and PS). For soils with corrected S and aluminum levels, broadcast ES may be more appropriate to provide sugarcane nutrition in the medium- or long-term use. Long-term studies on the continuous application of S sources on soil and crop yield would help refine S management strategies to improve fertilizer use efficiency across sugarcane-producing regions under different edaphoclimatic conditions.

### 5 | CONCLUSIONS

On average, sugarcane was responsive to S fertilization and its residual effects across the sites. Positive effects were observed at two sites (I and II) in the first ratoon cycle, and positive residual effects were observed at one site (III) in the second ratoon cycle. Yield gains due to S sources application averaged 5–7 Mg ha<sup>-1</sup> in the first ratoon cycle and 7–9 Mg ha<sup>-1</sup> in the second ratoon cycle (residual effect). There was no significant difference in yield between the 50 and 100 kg S ha<sup>-1</sup> application rates. The positive effect of ES application was greater in the second ratoon cycle (residual effect) than in the first ratoon cycle, indicating its potential for medium- and long-term use.

The band application of PS at 100 kg S ha<sup>-1</sup> increased leaf S concentration in both ratoon cycles, and the broadcast application of ES and gypsum at 50 kg S ha<sup>-1</sup> improved leaf S concentration in the second ratoon cycle compared to other sources. Across all sites, soil S concentration in the 0–25 cm layer 12 months after S application was significantly higher at 100 kg S ha<sup>-1</sup> than at 50 kg S ha<sup>-1</sup>. The highest soil S concentrations were observed in the gypsum treatments. However, soil S concentration in the surface layer was less than 10 mg dm<sup>-3</sup> at most sampling timepoints, indicating the need for annual S fertilization in ratoon sugarcane cycles. Soil S levels should be regularly monitored to ensure adequate levels and sugarcane yields. Combining sulfate and ES sources could provide both short- and long-term S availability in sug-

arcane ratoon areas with straw coverage and sulfur-deficient soils.

## AUTHOR CONTRIBUTIONS

**Rafael Otto:** Conceptualization; data curation; funding acquisition; investigation; methodology; project administration; resources; supervision; validation; writing—original draft; writing—review and editing. **Lucas Miguel Altarugio:** Data curation; formal analysis; investigation; methodology; validation; visualization; writing—original draft; writing—review and editing. **Victor Xavier Rizzo:** Investigation; methodology; visualization; writing—original draft. **Estêvão Vicari Mellis:** Investigation; methodology; validation; visualization; writing—original draft. **Sarah Tenelli:** Data curation; formal analysis; investigation; validation; visualization; writing—original draft; writing—review and editing. **Johnny Rodrigues Soares:** Data curation; investigation; methodology; validation; visualization; writing—original draft; writing—review and editing. **Sarah Mello Leite Moretti:** Conceptualization; data curation; investigation; methodology; validation; visualization; writing—original draft; writing—review and editing.

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## ORCID

Rafael Otto  <https://orcid.org/0000-0003-1472-298X>

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