EVALUATION OF SULFUR SOIL TESTS FOR MONTANA

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ABSTRACT

There are no sulfur (S) fertilizer rate guidelines in Montana due to inconsistent yield responses, high S soil levels in many regions, a minimal number of S fertility trials in the state, and the lack of Montana and regional data that identify the best soil S tests to use. A study was initiated in 2023 to fill this research void. Three crops (spring canola, pea, and wheat) were grown at three sites, and fertilized with 0, 7.5, 15, and 30 lb S/ac using two S sources (potassium sulfate, gypsum) in a randomized complete block design (four replicates). Treatments with S increased yield above 0S controls in wheat at two sites (by 8 to 15 bu/ac) and in canola at one site (by 18 bu/ac). There was no yield benefit above 7.5 lb S/ac, and no other yield responses were observed. Soil S was extracted from soils collected pre-seeding (0-6, 6-12, and 12-24 in.) with 8 mM monocalcium phosphate analyzed with ion chromatography (Caphos IC) and inductively coupled plasma-optical emission spectrometry (Caphos ICP), 0.12M KCl (turbidimetric) and Mehlich3 extract with ICP. There generally were strong relationships between the summed S pools (lb ac^{-1}) in the 0S controls for each depth (0-6, 0-12, 0-24 in.) and mid-season foliar S for each crop, suggesting S test levels were related to S availability. When comparing S uptake for all crops in the lowest S soils with soil S pools, there were no relationships in the 0-6 in depth and only turbidimetric S was related to S uptake in the 0-12 in. depth ($p<0.1$). For the 0-24 in. depth, S uptake was related to S pools using Caphos IC (R^2=0.39), Caphos ICP (R^2=0.64), and turbidimetric ($R^2 = 0.72$) suggesting that Caphos ICP and turbidimetric S tests can adequately assess S availability if the soil is sampled deep enough. Given that sulfate is a mobile nutrient, we propose developing S rate guidelines in lb S bu^{-1}.

INTRODUCTION

Soil S tests are generally not predictive of yield responses to S fertilization for a variety of reasons (Franzen, 2018,) including high reliance of crops on in-season S mineralization (Goh & Pamidi, 2003; Carciochi et al., 2019) and high S spatial variability based, in part, on landscape position (Franzen & Grant, 2008). In semi-arid regions, there is also the possibility that calcium carbonate-coated gypsum particles (Keren and Kauschansky, 1981) or calcium carbonate-sulfate co-precipitates (Hu et al., 2005) could become available in lab extractions, but remain unavailable in dry, generally higher pH field environments. In addition, given the mobility of sulfate, it's possible that soil tests performed on the top 6 or 12 inches don't capture a majority of S available to crops, especially deep-rooted crops. Finally, it's possible that standard soil S tests do not adequately reflect S availability.

Although there have been several published studies comparing soil S tests with S availability and/or yield, relatively few of those have been conducted in the field, especially in the past two decades. In a greenhouse study on soils ranging from sand to loam, S levels for 10 of 11 extractants were strongly related ($R^2 > 0.70$) to S uptake, including 0.01M $Ca(H_2PO_4)_2$ (MCP), 1M ammonium acetate, and 0.5M $NaHCO_3$ (Arora and Sekhon, 1979). Zhao et al. (1994) found strong relationships between S uptake and S levels determined by 0.016M KH_2PO_4, 0.01M MCP, 0.01M $CaCl_2$, and water analyzed by either IC or ICP in pots, across soils ranging from loamy sand to clay loam. There were strong relationships between forage yields and soil S extracted with 0.25M

KCl heated at 40, 80, and 100°C (by ICP), 0.01M MCP (by ICP), and water (by IC), but no relationships with S extracted using 0.5M $NaHCO_3$ analyzed turbidimetrically or 0.25M KCl at 25°C in pasture soils (Blair et al., 1991). More recently published studies have compared soil S levels among different soil extraction and analysis methods (Ketterings et al., 2011; Rogers et al., 2019). Importantly to the western U.S., 0.01M MCP S concentrations (via ICP-OES) in Idaho soils (top 12 in.) were strongly related with both Mehlich-3 and Haney S concentrations, although Mehlich3 S concentrations were two to three times greater than MCP-extracted S concentrations when S was below approximately 10 mg S kg^{-1} (Rogers et al., 2019).

In the western U.S., the reference method for soil S involves extracting soil with 8 mM MCP, removing dissolved organics with activated carbon, adding $BaCl_2$ to form $BaSO_4$ followed by turbidity analysis with a spectrophotometer (Miller et al., 2013). There has been an apparent shift away from turbidimetric analysis by many laboratories, likely in part because it is time-consuming, and results can suffer from interferences with organic colloids and metals, and inconsistencies in $BaCl_2$ particle size, temperature, and standing time of the suspension (Ketterings et al., 2011). Another issue is that the turbidimetric method has a detection limit near 2 mg kg^{-1}, which over a 2 ft profile, represents 16 lb S ac^{-1}, near or above what many dryland western crops require, making the method too imprecise at low S levels. Analysis of MCP and other S extracts is becoming more commonly conducted by ICP, which would include both dissolved inorganic and organic S. This has the possible advantage that the ICP results could reflect S mineralization amounts but could also inflate S availability, especially if much of the dissolved organic S does not mineralize before the crop needs it. In New York soils, MCP-turbidimetric S concentrations in the upper 12 in. were approximately 10-15 mg kg^{-1} lower than MCP-ICP S concentrations (Ketterings et al. 2011), but we have not found similar comparisons in western soils.

Due to the lack of published studies in the western U.S. that have evaluated the ability of different soil S tests to predict crop responses of S fertilization, we initiated a field study in 2023 with the following objectives:

1. Determine yield and quality responses of S fertilization on spring canola, pea, and wheat.
2. Evaluate relationships between soil S levels from four S soil tests and plant available S.
3. Identify the sampling depth for soil S that best estimates plant S availability.

METHODS

Design. Spring canola, pea, and wheat were grown at Central Ag Research Center (CARC) near Moccasin, Agronomy Post Farm (PF) near Bozeman, and Western Triangle Ag Research Center (WTARC) near Conrad in 2023. Yellow mustard replaced canola at Bozeman in 2024 to protect the local canola breeding industry and minimize bird damage. Sulfur (S) treatments consisted of a 0S control, and 7.5, 15, and 30 lb S ac^{-1} of potassium sulfate (0-0-50-17) and gypsum (0-0-0-18), side banded at CARC and PF, and broadcast at WTARC, for a total of seven treatments with four blocks, in a randomized complete block design for each crop. Sulfur rates were halved in 2024 due to the lack of a yield benefit above 7.5 lb S ac^{-1}.

Soils. Soils were sampled pre-seeding by block (0-6", 6-12", 12-24" or to rock at CARC) and 24-36" at Post Farm only. Soils were extracted with 8mM MCP and analyzed for sulfate with ion chromatography (Caphos IC) and for total S with inductively coupled plasma (Caphos ICP) in MSU's Environmental Analysis Laboratory (EAL). Soils were also extracted with 0.12M KCl and analyzed for S on a spectrophotometer ("turbidimetric") and with a Mehlich3 extract using ICP at AGVISE Laboratories. The S concentrations in mg kg^{-1} were converted to lb S ac^{-1} by using bulk densities and summed across depths over the top 2 ft. (Table 1). Sulfate levels were very low at CARC, low at Post Farm, and medium to very high at WTARC. These results are consistent with previous work and correlate with high sulfate leaching potential at CARC and PF due to cobbly soils (CARC) and high precipitation/irrigation amounts (PF), and the presence of gypsum in many soils around Conrad resulting in high, and variable, available S levels. The Mehlich3 S levels were much higher than for the other three methods, likely because the Mehlich3 extract contains a strongly buffered acid and a chelate which could dissolve lime coatings on gypsum, and release more S bound in organic forms. In the top 6 in., soil pH at CARC, PF, and WTARC averaged 7.2, 7.4, and 8.0, respectively, and soil organic matter averaged 4.0, 2.9, and 3.5%, respectively. Soil textures in the upper 6 in. were clay loam at CARC and WTARC, and silty clay loam at PF. Soil test S results for 2024 are still being evaluated.

Table 1. Soil sulfur test levels in 2023 for each study site for the top 24 inches (or to rock at CARC) by summing 3 depths (0-6, 6-12, 12-24 in.) and averaging across 3 crops and 4 blocks. Range is shown in parentheses.

Soil S test	CARC	PF	WTARC
	------------------------------- lb S ac^{-1} -------------------------------		
Ca phosphate IC EAL	2.1 (1.4-3.1)	4.8 (3.6 – 6.2)	188 (14.6-705)
Ca phosphate ICP EAL	8.9 (6.1-13.2)	14.6 (10.7-16.9)	179 (28.4-573)
KCl turbidimetric AGVISE	13.0 (3.8- 0.7)	24.2 (12.0-30.6)	232 (35.0-526)
Mehlich3 AGVISE	61.1 (32.7 80.9)	77.6 (53.7-94.7)	525 (96.8-1492)

CARC- Central Ag Research Center; PF-Arthur Post Farm; WTARC-Western Triangle Ag Research Center; IC – ion chromatograph; ICP – inductively coupled plasma; EAL – MSU's Environmental Analysis Laboratory; AGVISE - AGVISE laboratories, Northwood ND and Benson MN.

Tissue S and S uptake. Tissue was collected from ~25 plants in the control and each K_2SO_4 plot in late June, by cutting the 5th leaf from the top on canola (at ~20% bloom), the most mature leaf in pea (at early bloom), and flag leaf in wheat (at flag leaf). Tissue was dried, ground, and analyzed for total S via combustion (LECO CNS 928). Tissue S levels in 2023 were highly responsive to S fertilization at CARC and PF, especially in canola (data not shown). Approximately one week before harvest, plants were cut at the soil surface (1-m of a row) from 0 and 15 lb S ac^{-1} treatments (at PF), dried, and threshed. Grain and the biomass remaining were weighed and analyzed for S to obtain an S harvest index (SHI). The grain from combine-harvested plots was also analyzed for S, and S uptake was calculated using SHI.

Economics. We assumed \$6 bu^{-1} wheat, \$15 bu^{-1} canola, and an S cost of \$0.40 lb^{-1} (which accounts for the N value, as urea, in ammonium sulfate).

RESULTS AND DISCUSSION

Yield. In 2023, S fertilizer increased canola grain yield by 18 bu ac^{-1} at CARC (with K_2SO_4), and wheat grain yield by 8 and 15 bu ac^{-1} at CARC and PF, respectively (data not shown). High soil S levels appeared to prevent any S responses at WTARC. There were no yield benefits of fertilizing

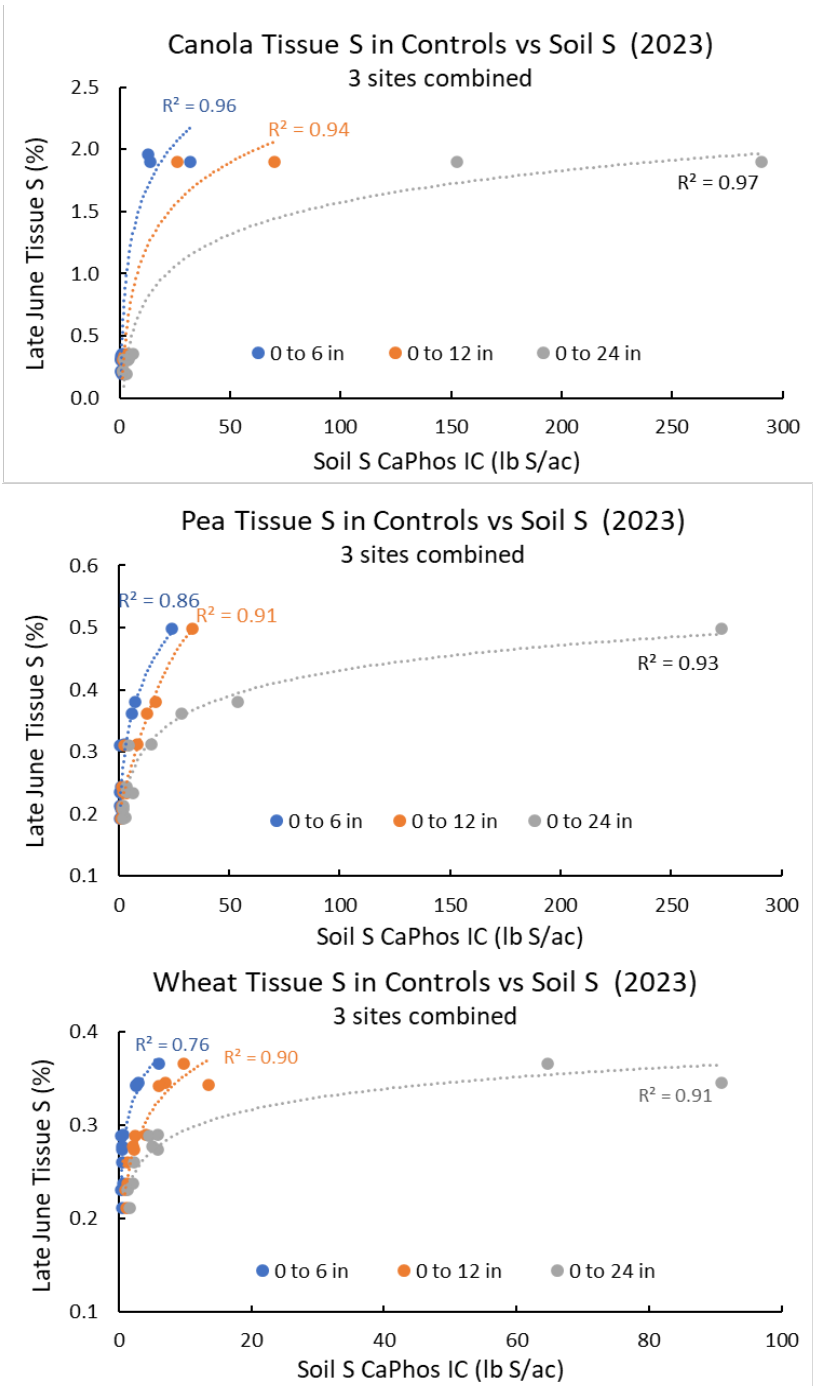

Figure 1. Relationship between leaf tissue S in 0S controls collected late June and Caphosphate S analyzed by ion chromatograph (IC).

with more than 7.5 lb S ac^{-1}. In 2024, there were no yield responses from S fertilization, likely due to less precipitation and a hotter July.

Evaluation of soil sampling depth and analytical method. Tissue S levels should be strongly related to soil S availability; therefore, tissue S levels in the 2023 controls were compared with soil S amounts for 0-6, 0-12, and 0-24 inch depths for Caphos IC, Caphos ICP, and turbidimetric S for each crop (Figure 1; Caphos IC only, as an example). There were strong relationships (mostly logarithmic) across soil depths between tissue S and Caphos IC S (R^2 = 0.76-0.97) and Caphos ICP (R^2=0.87-0.99; data not shown), and weak to strong relationships for turbidimetric S (R^2 = 0.31-0.93; data not shown). This finding suggests that soil S tests estimate S availability reasonably well. There should be a strong, and near 1:1 relationship between S uptake and available S (soil S + fertilizer S) when S levels are low (i.e. at CARC and PF) because plants should scavenge most available S. We determined aboveground S uptake in 2023 for each crop at PF and CARC (controls plus the 7.5 lb S ac^{-1} rate, when S responsive). For the top 6 inches, there were no relationships between S uptake and available S for any of the four S methods (data not shown). For the top 12 inches, the only relationship between S uptake and available S was for turbidimetric S (R^2=0.63). When the top 24 in. of soil S were compared with S uptake, relationships were relatively strong for both Caphos methods and the turbidimetric method, while there was no relationship between S uptake and

Mehlich3 S (Figure 2). The combined results suggest that soil should be sampled at least 12 in. deep for S analysis to best reflect S availability.

For the top 24 inches, slopes between S uptake and available S were slightly less than 1.0 for both Caphos methods and 0.62 for the turbidimetric method. Mehlich3 greatly overestimated S availability (meaning points were well to the right of the 1:1 line) and available S using Mehlich3 was not well correlated with S uptake. Of the two Caphos methods, the ICP method has a better likelihood of estimating true S availability because it includes dissolved organic S (which could become available through S mineralization), whereas the IC method does not.

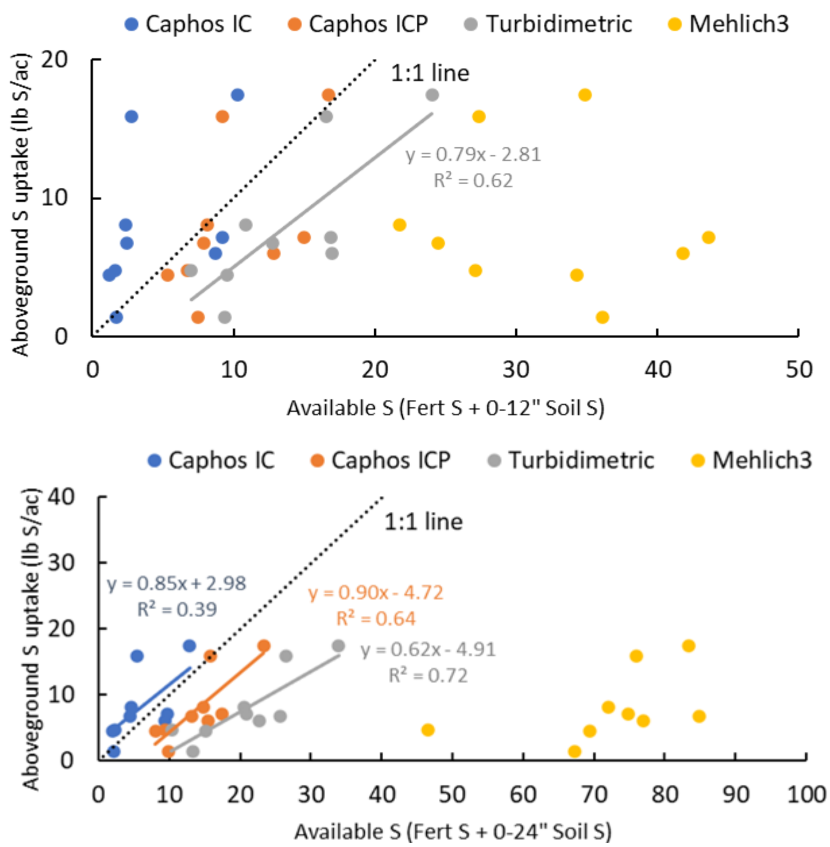

Figure 2. Relationships between aboveground S uptake and available S at CARC and PF in 2023 for controls of each crop and the 7.5 lb/ac treatment (as K_2SO_4) for the three S-responsive trials (canola at CARC; wheat at both CARC and PF). Upper – top 12-in. soil S; Lower – top 24-in. soil S.

Economic optimum S rates (EOSR). Guidelines for S fertilization are often based on a critical level even though sulfate is mobile, like nitrate, and N fertilizer rate guidelines are more often based on yield goals (e.g. 2.6 lb available N bu^{-1}). The only yield goal based on the S rate guideline we located was by the Canola Council of Canada (0.50 – 0.70 lb available S bu^{-1}). Notably, at CARC, the EOSR was 0.79 lb available S bu^{-1} when using Caphos ICP, slightly beyond the upper range of the Canola Council of Canada guidelines but was 1.39 lb S bu^{-1} using turbidimetric S (data not shown). The wheat EOSR was approximately 0.22 lb S bu^{-1} at both CARC and PF for Caphos ICP but ranged from 0.30 to 0.50 lb S bu^{-1} for turbidimetric S. We need more data to identify the optimum S test for MT.

Although we don't have enough data yet to have much confidence in establishing S guidelines based on yield goals for Montana crops, the low cost of S (~\$0.40 lb^{-1}, when considering the value of N in ammonium sulfate, \$1.00 lb^{-1} otherwise), indicates a relatively low economic risk of over-fertilizing compared to under-fertilizing. Specifically, gross revenue losses in 2023 controls were approximately \$50 to \$175 ac^{-1} for our three S-responsive trials (data not shown), compared to S costs of approximately \$3 ac^{-1} at the optimum S rate. Given the time and expense of conducting S fertility trials when there are often no yield responses, combined with the low cost of S, we propose that the ratio of EOSR to S uptake (in lb S bu^{-1} at S sufficiency) established in this and other S trials could be used to establish available S rate guidelines for other crops and possibly in other states within the region. Specifically, when using Caphos ICP 0-24 in. S pools, this ratio was approximately 2.5 for canola at CARC and 1.7 for wheat at CARC and PF based on canola aboveground S uptake of 0.32-0.34 lb S bu^{-1} and wheat S uptake of 0.13-0.15 lb S bu^{-1}.

CONCLUSION

In summary, deeper soil S pools (0-24 in.) were much stronger predictors of S uptake than surface pools (0-6 in.), a finding that was consistent across S tests. The Caphos ICP method (0-24 in.) showed the most promise at estimating S availability. After more site-years of data are collected from Montana, and possibly other western states, we hope to be able to establish S rate guidelines for common crops grown in the region.

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