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**Research Article** 

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# Effects of extra sulfur fertilization on soil pH and grain mineral concentration in wheat

# Bekir Atar<sup>1,\*</sup> 问

<sup>1</sup>Isparta University of Applied Science, Atabey Vocational School, Department of Plant and Animal Production, 32670, Isparta, Turkey

\*Corresponding Author: bekiratar@isparta.edu.tr

# Abstract

Increases in crop yield and decreases in industrial sulfur emissions reduce the amount of sulfur (S) in the soil. Sufficient S and proper pH play an important role in achieving the targeted yield and quality of wheat. In recent years, composite fertilizers containing sulfur and mineral substances have been produced and used in wheat production as fertilizers. In this study, an investigation was made as to whether the use of such fertilizer is sufficient for sulfur fertilization. Basal fertilizer  $(13.25.5 + 10 (SO_3) + Zn (0.5)) (250 \text{ kg ha}^{-1})$  was applied to the whole plot, and additional elemental sulfur was added. The amounts of sulfur in the plots at the end of the applications were 10, 300, 600 and 900 kg ha<sup>-1</sup> S. One year later, the high dose (600 and 900 kg ha<sup>-1</sup>) S resulted in a decrease in pH of about 0.5 pH units, while two years later, all S doses resulted in a 1 pH unit decrease. However, the changes in the second year were not induced by the S treatments. Accordingly, no significant effect of S doses on grain protein, mineral content or yield was determined. Wheat variety and year had a significant effect on grain nutrient content. The lowest sulfur dose (10 kg ha<sup>-1</sup> S) used in the experiment can be said to be sufficient for yield and mineral concentration.

Keywords: Wheat mineral concentration, Sulfur fertilization, Soil pH

# Introduction

Sulfur has long been known to be an essential element for higher plants (Duke and Reisenauer, 1986). It is used in plant protein, amino acid and enzyme synthesis (Scherer, 2001). Sulfur fertilizer and emissions of S are the main sources of S in the soil. In the last decades, sulfur emissions in Europe (Hicks et al., 2002) and the use of sulfur fertilizer have decreased significantly. Although there was a fluctuation between 1990 and 2015, there has been no significant reduction in sulfur emissions in Turkey (Anonymous, 2017). The amount of sulfur in the soil decreases due to increased yield, low S emission, intensive agriculture, the use of low S content fungicides, stubble burning, etc. (Gupta et al., 1997). When the yield increases threefold, the amount of S removed from the soil approximately doubles (McGrath et al., 1996). In recent years, sulfur deficiencies have emerged in wheat production areas in Europe and many other parts of the world (Zhao et al., 1998; Tisdale et al., 1986; Inal et al., 2003).

The availability of nutrients such as iron and zinc depends on soil pH (Tisdale et al., 1993; Sönmez et al., 2008). Sulfur is applied to the soil in different doses to amend alkaline soil pH and to make some nutrients available (Modaihsh et al., 1989; Kaplan and Osman, 1998; Usta, 1995). There is a positive correlation between the availability of sulfur in the soil and wheat nutrient contents. It also increases bread quality (Randall and Wrigley, 1986; Rendig, 1986; Ryant and Hrivna, 2004). In recent years, basal fertilizers containing sulfur and micronutrients have been used frequently in Turkey. This study was carried out to determine the effect of basal fertilizer containing sulfur and the application of extra granular sulfur on soil pH and wheat nutrient concentration.

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**ORCID:** 10000-0002-1446-5699

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#### Materials and Methods

The experiment was conducted in the 2015-16 and 2016-17 growing seasons at the experimental farm of the Faculty of Agriculture of Isparta Applied Science University. Two wheat varieties were used, one for bread (Tosunbey) and one for durum (Ç-1252). In the first year, the trial area was divided into four main plots and basal fertilizer (13.25.5 + 10 (SO3) + Zn (0.5)) was applied to all plots at the rate of 250 kg ha<sup>-1</sup> (32.5 N, 62.5 P, 12.5 K and 1.25 kg ha<sup>-1</sup> Zn). In addition to the sulfur from the fertilizer, granular sulfur was applied to the other plots (2, 3, 4) at 300, 600 and 900 kg ha<sup>-1</sup> S. Ten days before planting, sulfur scattered on the soil surface was mixed (15-20 cm deep) by rotavator. Wheat sowing was performed on 15 October 2015

with a plot seed drill. The plots had six rows arranged with 20 cm between rows, and a row length of 4 m. In addition to the basal fertilizer, Ammonium Nitrate® fertilizer was applied in the spring at a rate of 47.5 kg ha<sup>-1</sup>N (Feekes 5).

In the second year of the experiment, sowing was performed on the same main plots, and S fertilization was not applied again. The sowing was carried out on 21 October 2016, and 60 kg ha<sup>-1</sup> P (TSP®) fertilizer and 32.5 kg ha<sup>-1</sup> N (Ammonium Nitrate®) fertilizer were applied. In the spring (Feekes 5), Ammonium Nitrate® fertilizer was applied at 47.5 kg ha<sup>-1</sup> N. Soil characteristics determined by analyzing the soil before the experiment are given in Table 1, and the climatic data of the growing periods are given in Table 2.

Table 1. Soil characteristics of the trial field

Clay%	Silt%	Sand%	pН	CaCO <sub>3</sub> %	OM%	Cu(ppm)	Mn(ppm)	Fe(ppm)	Zn(ppm)	N(%)	P(ppm)	K(ppm)
16.2	44.8	39.0	8.5	29.4	1.60	1.50	5.4	1.70	0.75	0.10	15.0	108

pH measurements were made in soil samples taken from soil depths of 30 cm in the main plots of the experimental field at specific intervals (Figure 1). Yield was determined by harvesting by hand. Wheat grain samples taken for nutrient concentration determination were washed and rinsed with pure water and dried in 65°C ovens for 2 days. The dried grain samples were milled and sieved through a 0.5 mm sieve. Wheat flour samples (0.5 g) were digested with an  $H_2SO_4$  / HClO<sub>4</sub> mixture

(4:1 by volume), and grain Ca, Mg, K, Fe, Cu, Zn and Mn concentrations were measured by Atomic Absorption Spectroscopy (AAS-Varian, FS 240) (Uygur and Şen, 2018). Phosphorus concentration was determined by a spectrophotometer (T-80) after adding vanadomolybdate coloring reagent. Nitrogen concentration was determined by the Kjeldahl method. The ANO-VA procedure was performed with SPSS software. Separation of the main effects was performed by Duncan test at p <0.05.

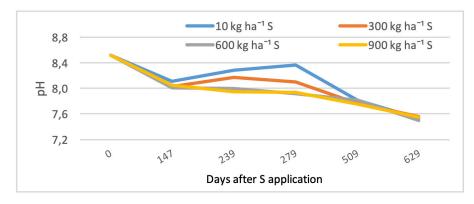
Table 2. The climate data of experimental years

Climatic factors	Years/ months	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Means /Total
	2015-16	14.6	9.1	2.5	1.3	7.3	7.6	14.0	14.6	21.6	25.0	11.8
Mean tempera-	2016-17	14.8	7.2	0.3	-0.8	3.0	7.3	10.6	14.9	20.1	25.2	10.3
ture (°C)	Long term	12.9	7.4	3.5	1.9	2.9	6.2	10.7	15.6	20.2	23.6	10.4
Total precipi-	2015-16	23.1	17.5	6.4	101.6	33.3	59.9	47.8	87.6	12.4	25.7	415.3
tation	2016-17	1.6	48.8	33.5	87.8	3.6	74.4	25.6	149.5	30.9	13.1	468.8
(mm)	Long term	38.0	46.3	84.9	72.2	64.7	54.2	56.0	51.4	29.8	14.6	512.1

#### **Results and Discussion**

The pH values of the soil samples taken from the plots at various times are given in Figure 1. At the beginning of the experiment, the pH value was 8.52. After five months of sulfur application (after 147 days), the pH of all sulfur doses had decreased by an average of 0.5 and was between 8.01 and 8.11. After application at a dose of 10 and 300 kg ha<sup>-1</sup> S in the second measurement and at a dose of 10 kg ha<sup>-1</sup> S in the third measurement, relative increases in pH were observed. At the second year harvest (after 629 days), pH had decreased by 0.20 and 0.32 degrees compared to the previous measurement (after 509 days). There was a 1 degree decrease in pH in all S doses compared to the beginning. During the whole trial period, fluctuations were observed at doses of 10 and 300 kg ha<sup>-1</sup> S, while

a steady decrease was observed at doses of 600 and 900 kg ha<sup>-1</sup> S. A high sulfur content in soil has the effect of oxidizing S with organic matter, and giving a high pH (> 6.5) (Zhao et al., 2015). The ability of applications of elemental S to reduce soil pH was seen in a previous study (Modaihsh et. al., 1989; Usta, 1995). The decrease of soil pH became more pronounced in the second year, but this decrease is likely to be a seasonal effect, with the basal fertilizer treatment also showing the same pH as the elemental S treatments. However, the difference after the second sampling may be explained in terms of the oxidation of S, which gives off some H ions, reducing soil pH as is evident in Fig 1. Since soil carbonate content is very high, the pH differences are not long lasting, and the treatment effect in the second year is not evident.



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Figure 1. The soil pH values

Wheat yield and nutrient concentrations of grain are given in Table 3. Nitrogen content (N) and protein ratio were found to be different between the years, with the second year (2.76%) higher than the first year (2.00%). The nitrogen content (2.09%) and 2.83%) of Tosunbey was higher in both years compared to C-1252 (1.92% and 2.69%), and the difference was statistically significant in the second year. Among the sulfur doses, the lowest N content was found at a 600 kg ha-1 S dose (1.78% in the first year, 2.64% in the first year), and the highest at a 900 kg ha<sup>-1</sup> S dose in both years (2.22% in the first year, 2.83% in the second year). Among the interactions, the lowest N rate was determined at the 600 kg ha-1 S dose and Ç-1252 cultivars (1.66% in the first year, 2.53% in the second year), while the highest N rate was at the 900 kg ha-1 S dose and cultivar C.1252 (2.27%) in the first year. In the second year, the highest N concentration was found at the 10 (basal fertilization) and 900 kg ha<sup>-1</sup> S dose and Tosunbey variety (2.91%). Although the highest protein ratio was found at a dose of 900 kg S, the protein ratio was not proportional to the S doses. Sulfur is a main component of protein, and S deficiency can limit the rate of grain protein (Westermann, 1993). Protein synthesis requires 1 part S per 15 parts N by weight, and lack of sulfur availability affects N assimilation rather than nitrogen uptake (Stewart and Porter, 1969; Freney et al., 1978; Sahota, 2006). Under normal yield conditions, it is necessary to apply one tenth of sulfur to the amount of nitrogen (Flowers et al., 2007). Considering the protein yield per decare in this study, it can be said that even the amount of S in the basal fertilizer (10 kg S ha<sup>-1</sup>) is able to sustain the protein synthesis without any adverse effect.

It does not seem possible to make a positive or negative interpretation of the effect of sulfur doses on grain nutrient contents. Rasmussen et al. (1975), Zhao et al. (1999), and Ercoli et al. (2012) stated that sulfur uptake and assimilation vary depending on the N/S ratio in the soil, irrigation, and amount and time of S and N fertilization. In both years of our study, the lack of precipitation in the period of germination and the irregular distribution of precipitation during the growing period limited the vegetative development and yield of the wheat. This in fact negatively affected nitrogen efficiency and the yield. S has a synergistic effect with nitrogen (Rossini et al., 2018), and can be expected to have no effect on the content of other nutrients under low nitrogen use efficiency and yield. Zinc can be transported to the grain by forming ligands with S-containing amino acids (Haydon and Cobbett, 2007; Torrance et al., 2008). Concentrations of zinc and other nutrients did not show significant changes with different sulfur doses. This shows that even the lowest sulfur dose used  $(10 \text{ kg ha}^{-1})$ is sufficient in terms of nutrient content at these yield values. Grain nutrient concentrations (N, P, K, Mg, Cu, Zn and Mn) were significantly higher in the second year. In the first year, only Fe concentration was found to be high, and in fact there may be an effect of S induced pH changes in such behaviour. As the yield increases, the concentration of minerals in the grain decreases (Graham et al., 1999; Garvin et al., 2006) in relation to the dilution effect, and grain quality is affected by climatic conditions (Ducsay and Lozek, 2004). High grain nutrient concentrations in the second year of the experiment can be explained by the decrease in yield. Grain nutrient concentrations of Tosunbey were higher than those of C-1252, except for potassium. There are wide variations between varieties and species in terms of nutrient concentrations (Liu et al., 2006; Zhao et al., 2009; Kara, 2013; Uygur and Şen, 2018).

No significant difference in yield was found between cultivars. Although grain yields were quite low in both years, the second year value (1202 kg ha<sup>-1</sup>) was lower than the first year (1893 kg ha<sup>-1</sup>). The first trial year (October 2015, November 2015 and December 2015) and second trial year (October 2017) had well below the average amount of rainfall (Table 2). This had a negative effect on the germination rate, and was the main reason for the low yield. Sulfur doses had no effect on yield in the first year, but yield increased in parallel with increasing sulfur doses in the second year. The yield was lowest in the control 10 kg ha<sup>-1</sup> S (1012 kg ha<sup>-1</sup>) and was highest at 900 kg ha<sup>-1</sup> S (1485 kg ha<sup>-1</sup>). The wheat plant removes 15-25 kg S per hectare and needs 2-3 kg S to produce 1 ton of grain (Zhao et al., 1999). Inal et al. (2003) stated that sulfur fertilization of 20 kg ha<sup>-1</sup> may be sufficient to reach sufficient yield in bread and durum wheat varieties. However, Zhao et al. (1999) stated that this dose (15-25 kg ha-1) should be increased to increase the yield. This explains S doses not having an effect on the yield in the first year, but contrasts with the yield values in the second year. Environmental factors were likely to have determined the yield in the first year, and it shadowed the main effect of S fertilization on yield.

Bekir Atar

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Table 3. Wheat grain mineral concentrations and yield at different sulfur doses (different letters indicate a statistical significance ns: not significant)

		(unitered	nt letters	muic		alist	ical sig	mine	ance,	115.	not sig	sinne	Jant)					
		N %	P mg kg <sup>-1</sup>					K mg kg <sup>-1</sup> C				Ca	mg kg-1		Mg mg kg <sup>-1</sup>			
Sulfur	Variety	2015-16 2016-17	2015-1	2016-17		2015-16		2016-17		2015-16		2016-17		2015-16		2016-17		
101 1 1	Tosunbey	2.23 ab 2.91	a 2474	f	3499	с	2840	cd	3966	а	347	b	446	ab	916	а	1271	а
10 kg ha <sup>-1</sup>	Ç-1252	1.81 ef 2.61	cd 2926	cd	4680	а	2665	de	3896	а	292	с	379	cd	833	bc	1153	bc
200 leg had	Tosunbey	2.05 bcd 2.75	bc 3076	ab	3382	c	2276	f	3554	b	413	а	419	b	953	а	1088	cd
300 kg ha <sup>-1</sup>	Ç-1252	1.95 cde 2.86	ab 2995	abc	2643	d	3335	b	3914	а	290	с	353	d	815	cd	1082	cd
(00 les heat	Tosunbey	1.90 de 2.75	b 2946	bcd	3208	с	2580	e	3531	b	404	а	377	cd	930	а	1206	ab
600 kg ha <sup>-1</sup>	Ç-1252	1.66 f 2.53	d 2654	e	2742	d	2415	ef	3948	а	308	с	370	cd	782	d	1038	d
0001 1 1	Tosunbey	2.17 abc 2.91	a 3081	а	4292	b	3841	а	3839	а	367	b	453	а	930	а	1300	а
900 kg ha-1	Ç-1252	2.27 a 2.74	bc 2858	d	4288	b	3007	с	3938	а	295	с	389	с	871	b	1081	cd
Mean		2.00 B 2.76	A 2876	В	3592	А	2870	В	3823	Α	339	В	398	А	879	В	1152	А
St Er.		0.20 0.10	211		733		506		197		49		37		64		105	
	10 kg ha-1	2.02 ab 2.76	ab 2700	с	4989	а	2757	b	3930	ns	319	ns	413	ab	874	ns	1212	ns
G 16	300 kg ha-1	2.02 ab 2.80	ab 3035	а	3012	b	2805	b	3734		351		385	ab	884		1085	
Sulfur	600 kg ha-1	1.78 b 2.65	b 2799	b	2975	b	2497	b	3739		356		374	b	855		1122	
	900 kg ha-1	2.22 a 2.83	a 2969	ab	4290	a	3424	a	3888		331		421	а	900		1271 1153 1088 1082 1206 1038 1300 1081 1152 105 1212 1085 1122 1190 1216	
Variety	Tosunbey	2.09 <sup>ns</sup> 2.83	a 2894	ns	3595	ns	2885	ns	3723	b	383	a	424	a	932	а	1216	a
variety	Ç-1252	1.92 2.69	b 2858		3588		2856		3924	а	296	b	373	b	825	b	1088	b

		Fe mg kg-1		Cu mg		Zn mg kg <sup>-1</sup>				Mn mg l			Yield kg ha-1						
Sulfur	Variety	2015-16 2016-17		2015-16		2016-17		2015-16		2016-17		2015-16		2016-17		2015-16		2016-17	
10 kg ha <sup>-1</sup>	Tosunbey	42.3 a 26	.0 bc	4.4	а	6.1	ab	20.0	ab	31.0	а	39.2	а	43.2	а	1481	cd	911	d
10 kg na ·	Ç-1252	35.7 bcd 26	.0 bc	3.9	bc	5.0	ab	16.1	с	29.2	ab	27.8	b	40.0	b	2162	ab	1114	cd
200 1 11	Tosunbey	37.4 bc 27	.1 bc	4.4	а	6.5	а	16.3	с	24.9	d	39.0	а	41.8	ab	2011	abc	1048	cd
300 kg ha-1	Ç-1252	39.7 ab 25	.1 c	4.6	а	4.9	b	18.1	bc	26.0	cd	30.3	b	32.7	e	1368	d	1145	cd
(00.1 1 -1	Tosunbey	35.2 cd 35	.0 a	4.5	а	6.0	ab	21.6	а	28.1	bc	37.0	а	39.4	bc	1777	bcd	1265	abc
600 kg ha <sup>-1</sup>	Ç-1252	33.4 cd 28	.3 bc	3.6	с	5.9	ab	15.2	с	29.9	ab	26.5	b	35.6	d	2400	а	1164	bcd
000 1 11	Tosunbey	39.6 ab 29	.5 b	4.5	а	6.3	ab	21.7	а	32.1	а	37.4	а	40.3	b	2168	ab	1452	ab
900 kg ha <sup>-1</sup>	Ç-1252	32.1 d 29	.2 b	4.1	ab	6.1	ab	18.0	bc	27.8	bc	29.7	b	37.0	cd	1777	bcd	1519	а
Mean		36.9 A 28	.3 B	4.3	В	5.9	А	18.4	В	28.6	А	33.4	В	38.8	А	1893	А	1202	В
St Er.		3.8 3.4	1	0.4		0.9		2.8		2.7		5.4		3.5		427		235	
	10 kg ha <sup>-1</sup>	38.9 a 26	.0 b	4.1	ns	5.6	ns	18.0	ns	30.1	а	33.5	ns	41.6	a	1821	ns	1012	c
G 16	300 kg ha-1	38.5 ab 26	.1 b	4.5		5.7		17.2		25.4	b	34.6		37.2	b	1689		1097	bc
Sulfur	600 kg ha-1	34.3 b 31	.7 a	4.1		5.9		18.3		28.9	а	31.7		37.5	ab	2088		1215	b
	900 kg ha-1	35.9 ab 29	.3 ab	4.4		6.2		20.0		29.9	а	33.5		38.7	ab	1972		1485	a
Variaty	Tosunbey	38.6 a 29	.4 <sup>ns</sup>	4.5	а	6.3	а	19.9	а	29.0	ns	38.2	a	41.2	a	1859	ns	1169	ns
Variety	Ç-1252	35.2 b 27	.2	4.1	b	5.5	b	16.9	b	28.2		28.6	b	36.3	b	1927		1235	

# Conclusions

Basal fertilizers containing sulfur and micro nutrients have been widely used in wheat production. It can be said that the amount of sulfur (10 kg ha<sup>-1</sup> S) in the basal fertilizer (13.25.5 + 10 (SO<sub>3</sub>) + Zn) or any other fertilizer containing S are sufficient in terms of yield, protein and nutrient content. There is no need for extra granular sulfur applications in wheat production. However, monitoring of S levels in soil and plants in the coming years would have beneficial features to guarantee sustainable yield and protein.

# Compliance with Ethical Standards Conflict of interest

The author declared no potential conflicts of interest with respect to the research, authorship, and publication of this article.

# Author contribution

The author read and approved the final manuscript. The author verifies that the Text, Figures, and Tables are original and that they have not been published before.

#### Ethical approval

Not applicable.

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# **Data availability** Not applicable. **Consent for publication** Not applicable.

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