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Determination of the Most Suitable Method to Predict the Available Sulfur Content in Cotton Growing Soils: Evidences from Aegean Coast, Türkiye

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A B S T R A C T

In this study, conducted on the selection of the most suitable method of determining the available sulfur content of soils where cotton is grown in the Coastal Aegean Region of Türkiye, soil and leaf samples were taken from a total of 40 cotton plantations in five different locations in the Coastal Aegean Region and Gediz Basin. Various physical and chemical characteristics of the soil's samples, which were taken from a depth of 0-30 cm, were determined, and the available sulfur contents were determined with the use of seven different extraction solutions. The extraction method or methods by which the highest correlation or correlations were obtained between the available sulfur content of the soils and leaf sulfur contents of the plants in the same plantation were assessed as the most suitable methods. According to the results of the study, the highest soil-leaf correlation was obtained by soil extraction with $Ca(H_2PO_4)_2$ solution, followed, in order, by the NH_4OAc, KH_2PO_4, cold water and NaCl methods. No significant correlation was found between the amounts of sulfur determined by extraction with $CaCl_2$ and KCl solutions and leaf sulfur contents. It was concluded that the most suitable methods for the determination of available sulfur in the soils of the Aegean Coastal Region where cotton is grown were the extraction methods using $Ca(H_2PO_4)_2$ and NH_4OAc solutions.

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Introduction

Sulfur is an essential nutrient for crop production. However, in recent years, sulfur deficiency has been recognized as a constraint on crop production all over the world (Eriksen et al., 2004; Girma et al., 2005; Schonhof et al., 2007; Mascagni et al., 2008). The main reasons are the reduction of sulfur dioxide emission from power plants and various industrial sources, the increasing use of low-S-containing fertilizers, the decreasing use of S-containing fungicides and pesticides, and high-yielding varieties (Scherer, 2001; Eriksen et al., 2004). Current inputs of S from atmospheric deposition are less than 10 kg ha^{-1} in most Western European countries (Hu et al., 2005), which is less than the amounts of S required by most crops (McGrath et al., 2002).

In plants, sulfur plays a crucial role in the synthesis of methionine, cystein and cystine amino acids, proteins, chlorophyll, and certain vitamins (Zhao et al., 1997; Havlin et al., 2004; Tiwari and Gupta, 2006). It is also known to be involved in the metabolism of carbohydrates, proteins and oils, the formation of cell walls and in flavor imparting compounds (Marschner, 2012).

In soils, S occurs in inorganic and organic forms, and is cycled between these forms via mobilization, mineralization, immobilization, oxidation, and reduction processes. While organic sulfur compounds are largely immobile, inorganic sulfur is more mobile, and sulfate (SO_4^{-2}) is the most mobile (Scherer, 2001). Sulfur is mainly taken up by plants via the roots from the soil solution as the divalent anion SO_4^{2-}. The major site of SO_4^{2-} uptake is the root hair region (Cacco et al., 1980)

Sulfate is the most common form of inorganic S and can be divided into SO_4^{2-} in soil solution, adsorbed SO_4^{2-} and mineral sulfur (Barber, 1995). Sulfur may precipitate in the form of SO_4^{2-} as calcium, magnesium or sodium sulfate. SO_4^{2-} also occurs as a co-crystallized or co-precipitated impurity with $CaCO_3$ and is an important fraction of the total S in calcareous soils (Tisdale et al., 1993).

As well as having high agricultural production value, cotton is an important source of income for many sectors because it is a crop with high industrial inputs. According to data from 2019/20, the Aegean Region is one of the important centers of production with 180 000 tonnes, which is 8.2% of total production of Türkiye where the 2.2 million tons of raw cotton produced on 478000 ha of land (TÜİK, 2020).

Improving quality and amount of yield obtained per unit area is directly related with botanical, environmental, cultural and economic factors, and also the amounts of available nutrients in the soil. Sustainability in agriculture can be ensured by determining the available amounts of nutrient elements in the soil, supplementing deficient amounts with fertilizers, and maintaining them throughout production. For this reason, it is of great importance to determine the amounts of available nutrient elements in the soil, using correct methods. The basic methods used to determine the amounts of available nutrients in the soil are laboratory, greenhouse and field methods. Because of time, workforce and economic disadvantages of the greenhouse and field methods, laboratory methods (extraction methods) are widely used in determining amounts of nutrients which are beneficial to the plant.

Various studies conducted in different ecologies have shown that $Ca(H_2PO_4)_2$, KH_2PO_4, NaCl and $CaCl_2$ solutions can be used successfully in the determination of available sulfur contents of soil (Fox et al., 1964; Rehm and Coldwell, 1968; Spencer and Freney, 1960; Amin, 1972; Çelebi, 1977; Kadakal, 2013).

However, use of a single method to determine available sulfur is not recommended because of the different soil types occurred with climatic and ecological differences. Moreover, the methods should be specific enough to provide detailed information about S availability for regional and plant basis. Thus, the aim of this study is to show in connection with soil-plant relations the most suitable extraction method or methods for use in determining the amounts of available sulfur in soils in the Aegean Region of Türkiye, where cotton is widely grown. The novelty of this study is that such a study had not been performed on soils where cotton plants of high economic value for the Aegean region are grown, and that it can illuminate different studies in similar ecologies.

Material and Method

The research was conducted at five different locations between the Aegean coast and the Gediz basin, where cotton is widely grown. The research material consisted of a total of 40 soil samples taken at a depth of 0-25cm from the districts of Koçarlı (8), Nazilli (6), Salihli (7), Söke (9) and Menemen (10), and leaf samples taken from the Nazilli-84 cultivar of cotton from the same plantations (Jackson, 1958; Reuter and Robinson, 1997). Cotton seeds were sown at the density of 7000-7100 plants/da, at a spacing of 15 cm within the row, and 75 cm between the rows. Before sowing, 200 kg ha^{-1} 20-10-10 (N-P-K) were applied to whole fields examined in the study. At the stage of blooming, once again 200 kg ha^{-1} 20-10-10 (N-P-K) were applied. The plantations were irrigated five times during the season at 15-20-day intervals by the furrow irrigation.

Approximately 100 leaf samples were taken at the first bud formation nearly 45 days after sowing (15 July), consisting of the third and fourth leaves from the growing point, together with their stems (Reuter and Robinson, 1997). The leaf samples were prepared for analysis by first washing them with tap water and distilled water, drying them at 65-70°C in a drying oven, and then grinding them. The amounts of sulfur in solutions obtained by the dry ashingmethod (reducing to ash at 550°C and dissolving in 3 N HCl) were determined by ICP-AES (VARIAN) (Shan et al., 1997).

The soil samples were air-dried and passed through a 2 mm sieve for physico-chemical analysis. The sand, clay and silt contents of the soil were determined by the hydrometric method (Bouyoucos, 1962). To determine pH and total content of water-soluble salt, the soil was saturated with water and the paste was tested with a pH meter with glass electrodes and a conductometer (U.S. Soil Survey Staff, 1951). Lime contents were determined with a Scheibler calcimeter, and amounts of organic matter were determined by the wet burning method with $K_2Cr_2O_7$ and H_2SO_4 (Reuterberg and Kremkurs, 1951). Nutrient elements in the soil samples were determined as follows: total nitrogen by the modified Kjeldahl method (Bremner 1965), and amounts of available K^+, Ca^{++}, Na^+ and Mg^{++} by AAS (VARIAN SpectraAA 220) in the filtrate after extraction with 1 N NH_4OAc. Available sulfur contents of the soils were determined by ICP-AES (VARIAN) in the extracts obtained by filtration by mixing 10 gram samples with seven different extract solutions (H_2O, KH_2PO_4-500 ppm, NaCl 1%, NH_4OAc-1.0 N, $Ca(H_2PO_4)_2$-500 ppm P and KCl) at a proportion of 1:5, and with a 15% $CaCl_2$ solution at a proportion of 1:6.6, and shaking for 30 minutes (Spencer and Freney, 1960; Ensminger, 1954; Williams and Steinbergs, 1959; McClung et al., 1959; Fox et al., 1964; Maynard et al., 1987).

Data obtained relating to various physical and chemical characteristics of the soil and leaf sulfur contents by methods used to determine the amounts of available sulfur in the soils were first subjected to Kolmogorov-Smirnov test for normality using the IBM SPSS 25.0 software. The correlations between variables were determined with the Pearson's correlation test. In the correlation analysis, data obtained by $Ca(H_2PO_4)_2$ extraction and in which the highest soil-plant correlation was determined was used.

Results and Discussion

The descriptive analysis of various soil characteristics and S contents available by different extraction methods of 40 soil samples are shown in Table 1 and Table 2 respectively. When the parameters were evaluated in terms of skewness, pH and available magnesium were shown to be negatively skewed, while the other parameters showed a positively skewed distribution (Table 1). When the available SO_4^{-2} content of the soils was evaluated, only the extraction method by KCl showed a negatively skewed distribution (Table 2).

The positively skewed distribution feature indicates that most of the values for the relevant parameters were below the mean (Köksal, 2002). These results showed that, among the soil properties and leaf sulfur contents examined, only pH was low, while the percentage of total nitrogen, available calcium and magnesium contents were medium, and the other parameters showed high variability, as seen in Tables 1 and 2.

Table 1. Descriptive statistics of physical and chemical characteristics of the research area

Soil Characteristics		Min.	Max.	Avg.	Standard Error	CV*	Skewness	Kurtosis
pH		6.95	7.93	7.62	0.19	0.02	-1.29	3.59
Total Soluble Salt	%	0.01	0.53	0.09	0.10	1.11	3.11	11.16
$CaCO_3$	%	1.00	22	10.73	6.43	0.60	0.18	-1.20
Organic Matter	%	0.05	3.10	1.04	0.71	0.69	0.78	0.55
Sand	%	10.82	77.12	40.98	19.51	0.48	0.42	-1.20
Clay	%	5.88	65.88	26.60	13.23	0.50	1.39	2.55
Silt	%	14.00	62.30	32.33	15.33	0.47	0.50	-1.28
Total N	%	0.05	0.12	0.08	0.02	0.22	0.18	-0.80
Available K	mg kg^{-1}	87	3675	401	544	1.36	5.87	36.09
Available Ca	mg kg^{-1}	969	5187	3742	756	0.20	4.03	-1.48
Available Mg	mg kg^{-1}	224	1210	741	223	0.30	-0.16	-0.13

*: Coefficient Variation <0.15: Low variability; 0.15-0.35: Average variability; >0.35: High variability (Wilding, 1985; Mulla and McBratney. 2000; Sağlam. 2013)

Table 2. Descriptive statistics of available S contents of soils in different extraction methods and sulfur content of leaves

Methods		Min.	Max.	Avg.	Standard Error	CV*	Skewness	Kurtosis
Pure water	mg kg^{-1}	0.70	195	24	42.85	1.82	2.81	7.61
KH_2PO_4	mg kg^{-1}	7.50	736	87	155.7	1.79	2.95	8.80
NaCl	mg kg^{-1}	17.0	645	103	154.8	1.50	2.57	5.91
NH_4OAc	mg kg^{-1}	0.60	134	15	26.20	1.79	3.66	13.85
$CaCl_2$	mg kg^{-1}	4.90	1236	126	261.0	2.07	3.22	10.21
$Ca(H_2PO_4)_2$	mg kg^{-1}	4.10	210	35	37.97	1.09	3.09	11.51
KCl	mg kg^{-1}	1.85	502	261	114.5	0.44	-0.75	1.33
Sulfur contents of leaves	%	0.36	1.76	0.87	0.37	0.43	1.22	0.77

*: Coefficient Variation <0.15: Low variability; 0.15-0.35: Average variability; >0.35: High variability (Wilding. 1985; Mulla and McBratney. 2000; Sağlam. 2013)

Classification of soil physical and chemical properties and nutrient contents according to limit values are given in Table 3.

The soils of the research area are loam and sandy-loam textured in general; all of them have a slightly alkaline reaction and have no risk of salinity. All the soils of the region have a slightly alkaline reaction (pH 7.5-8.5). The organic matter content of the soils examined was found to be generally low. In parallel with the low organic matter contents, in 75% of the soils' total N content was found to be inadequate. On the other hand, available Ca^{++} in 97.5% of the soils, and available K^{++} and Mg^{++} contents in all of them were adequate or high. Thus, Aydın et al. (2018), reported similar results concerning physical and chemical characteristics of cotton growing soils in the same area.

Available sulfur contents of the soils determined with different extraction solutions showed a wide variation according to the solution used (Table 2). The variability of the data obtained originates from differences in the extraction strengths of the solutions used (Spencer and Freney, 1960; Fox et al., 1964; Ensminger and Freney, 1966; Amin, 1972; Çelebi, 1977).

Ülgen et al. (1989) reported that the limit value of available sulfur (SO^{-2} - S), was 10 mg l^{-1} in the soils of Türkiye. However, they reported that according to the high yield obtained in relation to irrigation and plant species, at values below 15 and 20 mg l^{-1}, the amount of available sulfur in the soils may also be inadequate. Average available sulfur contents of the soils of the research area extracted by different methods varied between 15 and 261mg l^{-1}, and all of the soils examined were adequate with respect to amounts of available sulfur (Table 2). In different studies conducted in different ecologies, critical values for amounts of available sulfur in the soils were reported to be 10 mg l^{-1} extraction with phosphate solutions, and 14 mg l^{-1} extraction with acetate (0.5 N NH_4OAc + 0.25 N NaHOAc) and $CaCl_2$ (0.5%) solutions (Bansel et al., 1983, Singh et al., 2015). The available sulfur content of research area soils obtained with different extraction solutions were adequate according to the threshold values as formerly reported by the researchers (Table 2).

Available sulfur contents of the soils determined by seven different methods and relations between the sulfur contents of the leaves are given in Table 4.

When the Table 4 was examined the highest soil-plant correlation was obtained by the extraction of available sulfur content of the soils with $Ca(H_2PO_4)_2$ ($P \leq 0.01$). This was followed by extraction methods with NH_4OAc (1.0 N) ($P \leq 0.01$), KH_2PO_4, H_2O and NaCl solutions ($P \leq 0.5$), respectively. Similar to our findings, Çelebi (1977) reported that the highest correlation significant at a 1% level between available sulfur contents in the soils of the Antalya coastal region determined by different methods was obtained in extraction of the soils with a $Ca(H_2PO_4)_2$ (500 mg kg^{-1} P) solution, followed by extraction methods with KH_2PO_4 and H_2O. In a similar study conducted on the soils of the Meriç basin in the Trakya Region, Kacar et al. (1985) reported that the most suitable extraction solutions which could be used in the determination of available sulfur were KH_2PO_4 and 0.15% $CaCl_2$. In a study using different methods to determine the available sulfur contents of soil where Canola was grown in the Trakya region, Kadakal (2013) reported that the highest correlation between soil and plants was obtained in extraction of soils with $Ca(H_2PO_4)_2$ solution, followed by extraction with KH_2PO_4, $CaCl_2$ and NH_4OAc solutions.

Table 3. Some physical and chemical properties and nutrient content limit values of the soils (Zengin, 2012)

Soil Properties	Limit Value Range	Evaluation	%
Texture	-	Loam	22.5
		Sandy-Loam	22.5
		Sandy-Clay Loam	5
		Clay-Loam	20
		Clay	5
		Silty-Loam	2.5
		Silty-Clay	2.5
		Silty-Clayey-Loam	15
		Loamy-Sand	2.5
pH	7.5-8.5	Slightly Alkaline	100
EC $dS\ m^{-1}$	0-4	Non saline	100
	4-8	Slightly saline	-
	8-15	Moderately saline	-
	>15	Strong saline	-
$CaCO_3$ %	1-5	Low	22.5
	5-15	Moderate	45
	15-25	High	32.5
	>25	Very high	-
Organic Matter %	<0.5	Very low	27.5
	0.5-1.0	Low	20
	1.0-2.0	Medium	32.5
	2.0-3.0	High	7.5
	>3.0	Very high	5
Total N %	<0.045	Very low	-
	0.045-0.090	Low	75
	0.090-0.170	Sufficient	25
	0.170-0.320	High	-
	>0.320	Excessive	-
Available K mg kg^{-1}	50-140	Insufficient	5
	140-370	Sufficient	70
	370-1000	High	25
Available Ca mg kg^{-1}	< 238	Very low	-
	238-1150	Low	2.5
	1150-3500	Sufficient	27.5
	3500-10000	High	70
	>10000	Excessive	-
Available Mg mg kg^{-1}	< 50	Very low	-
	50-160	Low	-
	160-480	Sufficient	12.5
	480-1500	High	87.5
	>1500	Excessive	-

Table 4. Correlations between soil SO_4^{-2} contents determined by different extraction methods and leaf sulfur contents

Extraction Methods	$Ca(H_2PO_4)_2$	NH_4OAc	KH_2PO_4	H_2O	NaCl	$CaCl_2$	KCl
Leaf S content	0.503**	0.472**	0.378*	0.376*	0.373*	-0.024	-0.132

*: $P \leq 0.05$; **: $P \leq 0.01$

Ensminger and Freney (1966) reported that KH_2PO_4 and $Ca(H_2PO_4)_2$ extraction solutions could be used successfully in determining the available sulfur content of soils because they allowed the determination of a part of the SO_4^{-2} ions adsorbed along with SO_4^{-2} ions.

Tabatabai (1986) showed that $Ca(H_2PO_4)_2$, (KH_2PO_4), neutral salt solutions, hot water, NaOAc (pH:4,8) or $NaHCO_3$ extraction solutions could be used in determining sulfur which is available by plants in the soil. Also, it was reported that water-soluble SO_4^{-2} ions could be determined with $Ca(H_2PO_4)_2$ and (KH_2PO_4) solutions when a part of the sulfate ions adsorbed with passed into solution. Thus, it may be thought that obtaining the highest soil-plant correlation by the method of extraction with $Ca(H_2PO_4)_2$ in our study may originate from this mechanism.

The correlations between some physical and chemical characteristics and nutrient element contents of the soils and leaf sulfur contents were given in Table 5.

According to correlation analysis, negative correlations were observed between sand, silt (-0.736**) and clay (-0.611**) contents of soils. Similarly, negative correlations were also determined between pH and clay (-0.469**) and K contents (-0.480**). As is known, soil pH significantly influences plant growth by limiting the availability of nutrient elements (Akça et al., 2015).

Table 5. Relations Between Various Physicochemical Properties and Nutrient Contents of the Soils and S Contents of the Leaves

	Leaf S	Sand	Clay	Silt	pH	OM***	$CaCO_3$	EC	N	K	Ca	Mg	Soil SO_4^{-2}
Leaf S	1												
Sand	0.155	1											
Clay	0.005	-0.611**	1										
Silt	-0.199	-0.736**	-0.086	1									
pH	0.151	0.213	-0.469**	0.133	1								
OM*	-0.026	0.229	-0.108	-0.197	-0.056	1							
$CaCO_3$	0.093	-0.186	-0.118	0.335*	0.204	-0.178	1						
EC	0.174	0.029	0.041	-0.072	-0.177	-0.030	-0.020	1					
N	0.104	-0.267	0.244	0.128	-0.289	-0.404**	0.138	0.137	1				
K	-0.276	-0.058	0.232	-0.126	-0.480**	0.256	-0.023	0.206	0.277	1			
Ca	0.121	-0.081	-0.037	0.133	0.216	0.098	0.540**	0.034	0.061	0.344*	1		
Mg	0.084	-0.022	-0.084	0.100	0.179	0.130	0.531**	0.249	-0.021	0.376*	0.666**	1	
Soil SO_4^{-2}	0.503**	0.056	0.081	0.367*	-0.150	0.023	0.324*	-0.220	0.085	-0.020	-0.082	0.079	1

*: P≤0.05; **: P≤0.01, ***OM: Organic matter

From this point of view, relationships between pH and the available amounts of nutrient elements are reported by different researchers (Çelik and Katkat, 2005; Parlak et al., 2008; Turan et al., 2010; Karaduman and Çimrin, 2016; Bayram, 2019).

Strong positive correlations were determined between soil lime contents and available Ca (0.540**), Mg (0.531**) and $SO_4^{=}$ (0.324*) contents. Under the conditions of rising pH in parallel with high $CaCO_3$ contents of the soil, there is an increase in the amounts of basic Ca^{++} and Mg^{++} cations, which are important sources of soil alkalinity (Wahba et al., 2019). As a matter of fact, soil Ca, Mg (0.666**) and K (0.344*) contents, and the significant positive correlations found between Mg contents and K contents (0.376*), show that at high pH levels, basic cations predominate in soil solutions. Similar correlations have also been reported in studies conducted in different ecologies (Taşkın et al., 2015).

Conclusion

In this study, seven different extraction solutions were used to determine the available sulfur contents of the soils of the Aegean coast and the Gediz basin, which have an important potential for cotton production in the Aegean Region of Türkiye. Consequently, the most suitable method/ methods will be used to determine the available sulfur contents of research area's soils was exposed. Correlations between soil sulfur contents found by different methods and leaf sulfur contents showed that the most suitable method for the soils of the region was the method of extraction with a $Ca(H_2PO_4)_2$ solution. However, it was found that the method of extraction with NH_4OAc, which did not show a statistically significant difference from the $Ca(H_2PO_4)_2$ method and which showed a strong correlation, could also be recommended for the soils of the region. Also, it can be said that the method of extraction with NH_4OAc has an advantage in that it is the extraction solution used in the routine analysis method to determine the macro elements of soil. In addition, it is thought that this research can lead to a basis for research which conducted on different crops in different regions, where sulfur is of particular importance in growing industrial crops.

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