Türk Tarım ve Doğa Bilimleri Dergisi 11(2): 347–356, 2024
https://doi.org/10.30910/turkjans.1407307

TÜRK
TARIM ve DOĞA BİLİMLERİ
DERGİSİ

TURKISH
JOURNAL of AGRICULTURAL
and NATURAL SCIENCES

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Araştırma Makalesi

The Effect of Active Lime on the Availability of Metal Micronutrients in Narlı Plain Soils[¥]

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Geliş Tarihi: 20.12.2023 Düzeltme Geliş Tarihi: 26.03.2024 Kabul Tarihi: 30.03.2024

ABSTRACT

The low solubility of minerals containing metal micronutrients such as Fe, Zn, Cu, and Mn in alkaline or lime-rich soils adversely affects plant growth in these soils, leading to significant yield losses. This study was conducted to determine the effects of active lime on metal microelement availability in calcareous soils. The analysis of 55 soil samples taken from Narlı Plain in Kahramanmaraş Province revealed a significant positive correlation (r=0.95**) between active and total lime values. The study also indicated that an average of 58% of total lime was present in the active form. Furthermore, it demonstrated that as total lime increased, the proportional amount of active lime decreased. The points where the ratio of active lime to total lime is highest are observed at low total lime contents. As the total lime content increases, the ratio of active lime to total lime decreases. This is thought to be related to the measurement of relatively higher active lime contents as the calcium on exchangeable surfaces passes into solution when the total lime is low. Therefore, this situation calls into question the reliability of active lime analysis at low total lime contents. With the increase in total lime content, this relative effect disappears and more reliable results are obtained. While active and total lime values showed similar correlations with Mn and Cu, Fe showed a higher correlation with active lime than with total lime. Active lime did not exhibit superior predictability compared to total lime when explaining the availability of metal microelements, excluding iron.

Keywords: Active lime, total lime, micronutrients.

Narlı Ovası Topraklarında Metal Mikroelementlerin Yarayışlılığı Üzerine Aktif Kirecin Etkisi

ÖZ

Alkaline ya da kireçli topraklarda Fe, Zn, Cu ve Mn gibi metal mikro-elementleri içeren minerallerin çözünürlüklerinin düşük olması, bu topraklarda bitki gelişimini olumsuz etkilemekte ve önemli verim kayıplarına neden olmaktadır. Bu çalışma, kireçli topraklarda aktif kirecin ölçülmesinin metal mikro elementlerin yarayışlılıkları üzerine etkisini belirlemek amacıyla yürütülmüştür. Kahramanmaraş İli, Narlı Ovası'ndan alınan 55 toprak örneğinin analizleri, aktif ile toplam kireç değerleri arasında önemli pozitif korelasyon olduğunu (r=0.95**), toplam kirecin ortalama %58'inin aktif formda olduğunu ve de toplam kireç arttıkça aktif kirecin oransal olarak azaldığını ortaya koymuştur. Aktif kirecin toplam kirece oranın en yüksek olduğu noktaların düşük toplam kireç içeriklerinde olduğu görülmektedir. Toplam kireç miktarının artışı ile birlikte aktif/toplam kireç oranı düşmektedir. Bu durumun, toplam kirecin düşük olduğu durumlarda, değişebilir yüzeylerdeki kalsiyumun çözeltiye geçmesiyle göreceli olarak daha yüksek aktif kireç içeriklerinin ölçülmesi ile ilgili olduğu düşünülmektedir. Dolayısıyla, bu durum aktif kireç analizlerinin düşük toplam kireç içeriklerinde güvenilirliğini sorgulatmaktadır. Toplam kireç miktarlarının artışı ile beraber bu göreceli etkinin ortadan kalkması ile daha güvenilir sonuçlar elde edilmektedir. Aktif ve toplam kireç değerleri Mn ve Cu ile benzer korelasyonlar gösterirken, Fe toplam kirece göre aktif kireç ile daha yüksek bir korelasyon göstermiştir. Demir dışındaki metal mikro elementlerin yarayışlılıklarını açıklamada aktif kireç, toplam kireçten daha yüksek bir belirleyicilik sağlamamıştır.

Anahtar Kelimeler: Aktif kireç, toplam kireç, mikro elementler.

INTRODUCTION

The soils of Turkey are predominantly identified as calcareous. This situation is related not only to the sedimentary origin of the parent material but also to the inability of insufficient rainfall to wash lime from the profile, leading to the accumulation of lime in specific layers (Eyüpoğlu, 1999).

Micro nutrient deficiencies in soils are generally associated with high pH, lime, metal oxides, low organic matter, clay type, and clay content (Marschner, 1995). When soil pH exceeds 7, calcium (Ca^{2+}) ions are released, forming compounds with other ions in the soil (Karaman, 2012). In arid region soils, an excess of calcium antagonizes other nutrients, especially micronutrients. For instance, when there is an excessive amount of Ca in the soil, iron (Fe), potassium (K), and other elements are converted into forms that plants cannot utilize (Aktaş and Ateş, 1998). In calcareous soils, the high concentration of Ca^{+2} ions in the soil solution competes with Fe^{+2} ions for the same binding sites on clay surfaces, thereby inhibiting the uptake of Fe by plants (Machold, 1968).

Several factors in the soil diminish the efficacy of plant nutrients, and one of these factors is lime. Typically, soils in arid and semi-arid regions are calcareous. In fact, lime constitutes an important part of the soils of this region. The retention of manganese (Mn), zinc (Zn), and copper (Cu) in soils is directly related to alkaline pH, clay and silt particle size, active lime, and total lime. Active lime is generally associated with total lime and constitutes approximately 50% of the total (Estefan et al., 2013). Carter (1981) also reported that areas with active lime exceeding 7-9% experienced poor tree growth and leaf chlorosis. Active lime was identified as a more dependable field index value than total lime for diagnosing chlorosis.Numerous researchers have emphasized that determining the quantity of active lime, rather than total calcium carbonate, in the soil is crucial for assessing the availability of plant nutrients in alkaline soils. While the adsorption capacity of carbonate minerals, abundant in soils, may not be very high, they create favorable environments for precipitation reactions to occur, leading to the precipitation of metallic microelements in the form of carbonates, hydroxides, or hydroxy carbonates (Papadopoulos, 1989; Uygur, 2000; Mengel and Kirkby, 2001). This limitation hinders the effectiveness of the elements, even if their total amounts are sufficient. Consequently, deficiencies in microelements are frequently observed in calcareous soils (Güzel et al., 1991). Cox and Kamprath (1972) emphasized that total lime alone is not sufficient to indicate iron deficiency in soils. Loue (1986) emphasized that active lime should be measured as a measure of iron availability in soils. Reyes et al. (2006) also reported that they found relationships between chlorosis caused by iron deficiency in grapevines and soil pH, total and active lime. Active carbonate serves as an indicator of calcite reactivity and bicarbonate ions in soil solution, and it has been reported to be closely associated with iron deficiency in soils (Alcántara et al., 2000). Zinc is also one of the most deficient elements in calcareous soils, often limiting crop production (Navrot and Ravikovitch 1969; Navrot and Gal 1971). Saglam et al. (1997) also reported the Zn deficiency in a significant part of the Thrace region's soil. Plants benefit most from zinc under slightly acidic and neutral conditions. pH is an important factor affecting zinc availability. With an increase in pH, the availability of zinc decreases, and it precipitates as less soluble zinc hydroxides and zinc carbonates (Mengel and Kirkby, 2001). Manganese availability in soil varies significantly under the influence of numerous factors. With a significant decrease in pH, nearly all of the manganese in the soil is reduced to divalent manganese, increasing its solubility to toxic levels. Conversely, when there is excessive lime in the soil, manganese is found in an unavailable form for plants (Karaman, 2012). The mobility of Cu in soil solution also decreases as pH increases. The concentration of Cu in the soil solution of calcareous soils is quite low (Lindsay, 1972). The efficacy of active lime on micronutrient deficiencies in soils is a subject of debate. Therefore, it is essential to determine whether active lime is more effective than total lime in determining the availability of metal micronutrients in the soil.

The soils of the Narlı Plain in Kahramanmaraş exhibit variable lime contents ranging from 3.91% to 27.5%. It is also known that the pH levels of the region's soils are quite high (Saltalı et al., 2018). Therefore, attention should be paid to the availability of micronutrients in the soils of the region. Based on this, the present study investigated the effects of active lime and total lime content on the availability of trace metal micronutrients in alkaline soils taken from the Kahramanmaraş-Narlı Plain.

MATERIAL and METHOD

Experimental material for this study consisted of soil samples collected from Narlı Plain, situated approximately 30 km south of the city center of Kahramanmaraş Province. Soil samples were collected from 55 different locations at a depth of 0/30 cm. The samples were air-dried under natural conditions in a cool, dark environment, sieved through a 2 mm sieve, and then prepared for analysis. Salt determination was carried out using an electrical conductivity meter following the method outlined by Richards (1954), while pH determination was made in saturated soils according to Black (1965). Lime content was determined using a Scheibler calcimeter (Gülçur, 1974), and available microelements were analyzed by atomic absorption (Perkin Elmer) following extraction with the DTPA method, as outlined by Lindsay and Norwell (1978). The active carbonate method, developed by Drouineau (1942), relies on the principle of determining unreacted oxalate ($(NH_4)_2C_2O_4$) at the end of a 2-hour reaction of a soil sample with 0.1 M ammonium oxalate ($(NH_4)_2C_2O_4$), using titration with 0.1 M $KMnO_4$. The reactions that occur during the determination of active $CaCO_3$ are as follows (Loeppert and Suarez, 1996).

$$CaCO_3 + 2NH_4+ + C_2O_4^{-2} \rightarrow CaC_2O_4\ (k) + 2NH_3 + H_2O + CO_2\ \text{(Extraction)}$$

In this equation, oxalate reacts with soluble and exchangeable Ca, precipitating as calcium oxalate. Loeppert and Suarez (1996) reported that, therefore, the method should be referred to as the active Ca method instead of activate carbonate. The determination of active Ca or active carbonate involves titration of unreacted oxalate with Ca in an acidified extraction solution (addition of H_2SO_4) using potassium permanganate ($KMnO_4$). As shown in the reaction below, oxalate is converted to oxalic acid with the addition of sulfuric acid and reacted with MnO_4.

$$2MnO_4^- + 5\ H_2C_2O_4 + 6H^+ \rightarrow 2Mn^{+2} + 10CO_2 + 8H_2O\ \text{(Titration)}$$

As outlined in the method, 1 gram of each soil sample, sieved through a 1 mm sieve, was weighed and placed in centrifuge tubes. Subsequently, 25 ml of 0.1 M ammonium oxalate solution was added, and the mixture was shaken for 2 hours. Afterwards, 20 ml of the centrifuged samples were extracted and transferred to an erlenmeyer flask, followed by the addition of 5 ml of H_2SO_4. These samples were then incubated in a water bath heated to 70 °C for 30 minutes. At the conclusion of the incubation period, the samples were titrated with 0.1 N $KMnO_4$ until the color of the filtrates turned pink, and the values were recorded. The same procedure was conducted for the blank. Additionally, to determine the actual molarity of the potassium permanganate, the aforementioned steps were repeated using 0.2 g of sodium oxalate. The amount of active lime was calculated based on the obtained data using the following formulas *(where Vo represents the volume of $KMnO_4$ used in the blank titration (L), Vs is the volume of $KMnO_4$ reacting with the sample (L), C is the molar concentration of $KMnO_4$, DF is the dilution factor, and m is the amount of soil).*

$$M_{KMnO_4} = [Na_2C_2O_4(g)/\ 134\ g] * [1/\ KMnO_4\ (L)] * [2\ mol\ MnO_4\ /\ 5\ mol\ H_2C_2O_4]$$

$$\text{Active Lime (\%)} = (V_0\text{-}V_c)*(C)*(DF)*(5\ mol\ H_2C_2O_4/2\ mol\ KMNO_4)*(100gCaCO_3/mol)*100/m\ (g)$$

The descriptive statistics of soil samples were analyzed using the IBM SPSS 25 software package. The statistical relations of soil parameters were graphically represented using Microsoft Office 2016 Professional Plus Excel program.

RESULTS and DISCUSSION

Some Characteristics of the Study Soils

As a result of laboratory analyses conducted on soil samples from the study area, Table 1 provides the minimum, maximum, mean, standard deviation, skewness, and kurtosis values for pH, EC, active lime, total lime, active lime/total lime, and Fe, Zn, Cu, and Mn elements in the 55 soil samples.

Table 1. Descriptive statistics of measured values in soil samples

	N	Min	Max	Mean	SD*	Skewness	Kurtosis
pH	55	7.02	8.28	7.84	0.27	-0.948	0.790
EC	55	1.60	5.84	3.22	0.97	0.717	0.256
Active Lime	55	0.37	11.14	4.54	3.07	0.720	-0.772
Total Lime	55	0.56	37.09	11.71	10.66	0.473	-1.250
AL/TL**	55	0.23	1.93	0.58	0.34	1.993	4.527
DTPA-Fe	55	1.14	7.67	2.82	1.30	1.776	3.372
DTPA-Cu	55	0.50	1.87	1.21	0.32	3.404	14.016
DTPA-Zn	55	0.53	7.94	1.66	1.24	-0.157	-0.074
DTPA-Mn	55	3.63	38.96	13.63	8.47	1.358	1.425

*SD: Standard Deviation, **AL/TL: Active Lime/Total Lime

The analysis of soil samples collected from the research area revealed pH values ranging from 7.02 to 8.28, with an average pH value of 7.84 (Table 1). Furthermore, based on the classification values provided in Table 2 (Richards, 1954), it was observed that 20% of the soils fell within the neutral range (6.5-7.5), while 80% exhibited a slightly alkaline pH (7.5-8.5). When analyzing the electrical conductivity (EC) values of the soils, it was observed that the EC values ranged from 1.60 to 5.84 dS/m, with an average EC value of 3.22 dS/m (Table 1). According to the limit values provided in Table 2 (Maas, 1986), 85% of the soils were classified as moderately saline (2-4 dS/m), and 15% were classified as highly saline (4-8 dS/m).

Table 2. Soil pH and EC values and corresponding limit values *

pH	>4.5	4.5-5.5	5.5-6.5	6.5-7.5	7.5-8.5	>8.5
Class	Highly acidic	Moderate acidic	Slightly acidic	Neutral	Slightly alkaline	Highly Alkaline
EC	0-2	2-4	4-8	8-16	>16	
Class	Low	Moderate	High	Very high	Excess	

*(Richards, 1954), (Maas, 1986)

The total lime values exhibited a range from 0.56% to 37.09% and an average value of 11.71% (Table 1). Examination of the lime classification values reveals that 45% of the soil samples can be classified as moderately calcareous, while 20% are categorized as calcareous. Additionally, 25% of the samples exhibit a high degree of calcareous, and the remaining 10% are characterized by a very high level of calcareous (Table 3). Active lime values ranged from 0.37% to 11.14%, with an average of 4.54%. The ratio of active lime to total lime varied between 0.23 (23%) and 1.93 (193%), with an average of 0.58 (58%) (Table 1). Estefan et al. (2003) observed a comparable ratio of active lime value (50%) relative to total lime in their study. Upon examining these ratios, it became evident that the quantity of active lime can surpass the total lime amount, particularly when the total lime content is low. Therefore, it is suggested that using the term 'active Ca' instead of 'active lime' may be more accurate. Loeppert and Suarez (1996) also suggested that, in the active lime method, oxalate reacts with dissolved and exchangeable Ca, precipitating as calcium oxalate. Hence, the use of the term 'active Ca' is recommended.

Table 3. Limit values used in the evaluation of total lime in soil*

Lime (%)	Class
0-1	Slightly calcareous
1-5	Calcareous
5-15	Moderately calcareous
15-25	Highly calcareous
>25	Very highly calcareous

* Ülgen and Yurtsever, 1974.

When examining the DTPA-Fe values presented in Table 1, it is observed that the available Fe contents of the soils range from 1.14 to 7.67 mg/kg, with an average of 2.82 mg/kg. Based on the limit values provided in Table 4, it was determined that 87% of the soils were classified as insufficient, while 13% were considered sufficient. The observed quantities of available Cu in soils display heterogeneity, ranging from 0.50-1.87 mg/kg with an average concentration of 1.21 mg/kg. It was determined that all soil samples possessed adequate available Cu content. The observed data reveals that the quantities of available Zn in the soils exhibited a range from 0.53 to 7.94 mg/kg, with an average of 1.66 mg/kg. Based on the prescribed thresholds outlined in Table 4, it can be deduced that a majority of the soil samples (96%) were determined to be lacking in Zn. The Mn content in the soils varies from 3.63 to 38.96 mg/kg, exhibiting an average of 13.63 mg/kg. In accordance with the limit values delineated in Table 4, it was ascertained that all soils possess an adequate concentration of manganese.

Table 4. Limit values indicating the sufficiency levels of microelements in soil *

Micro nutrient	Suffiency level
Fe	>4.50
Zn	>0.76
Cu	>0.20
Mn	>1.20

* Lindsay and Norwell, 1976.

The graph below (Figure 1) illustrates a very strong correlation between total lime and active lime (r=0.94**), with the relationship being significant at the 1% level. This indicates a robust connection between total lime and active lime. Estefan et al. (2013) reported that active lime is typically related to total lime, constituting about half of the total lime. This relationship has also been observed by other researchers, including Carter (1981) and Ryan et al. (1981).

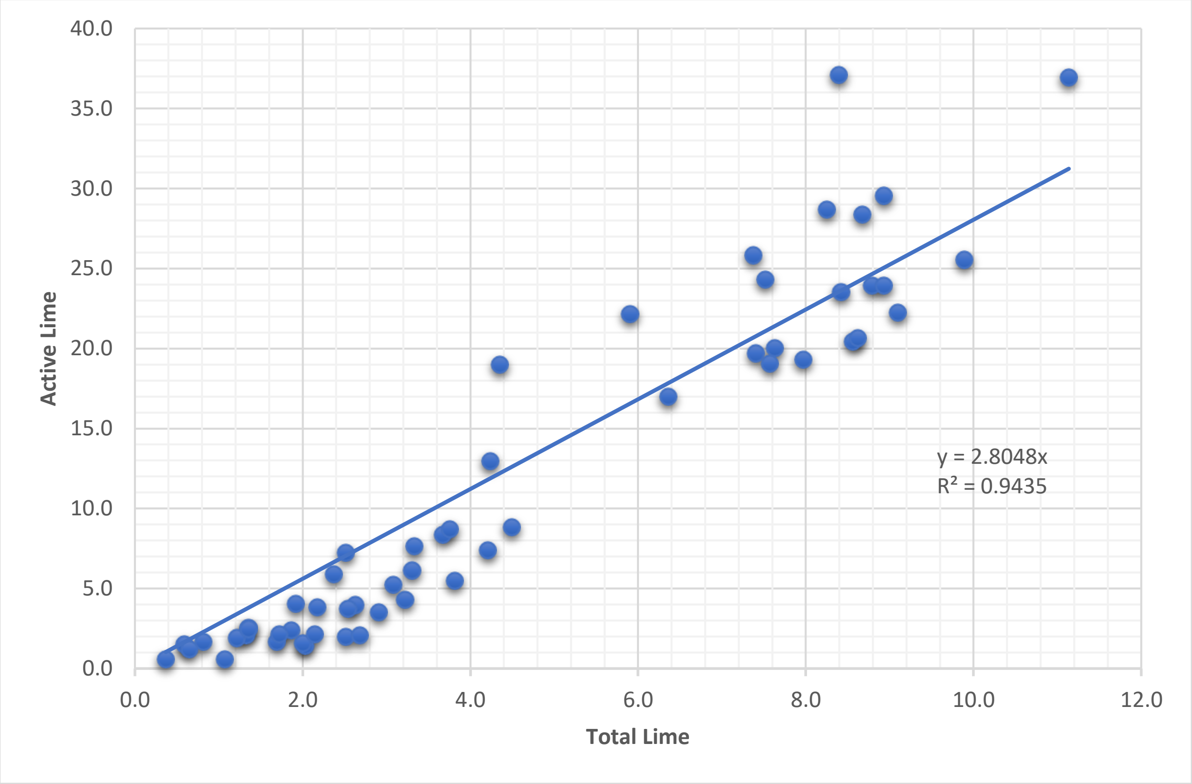

Figure 1. Relationship between active lime and total lime

The relationship between total lime and the active/total lime ratio is depicted in Figure 2. A negative correlation was observed between total lime and the mentioned ratio. Upon examination of Figure 2, it can be observed that the highest active lime/total lime ratio corresponds to the lowest values of total lime. As the total lime increases, the ratio decreases exponentially. Loeppert and Suarez (1996) reported that the reactive carbonate in soils could be determined acceptably, although not with complete accuracy, using the active carbonate method.

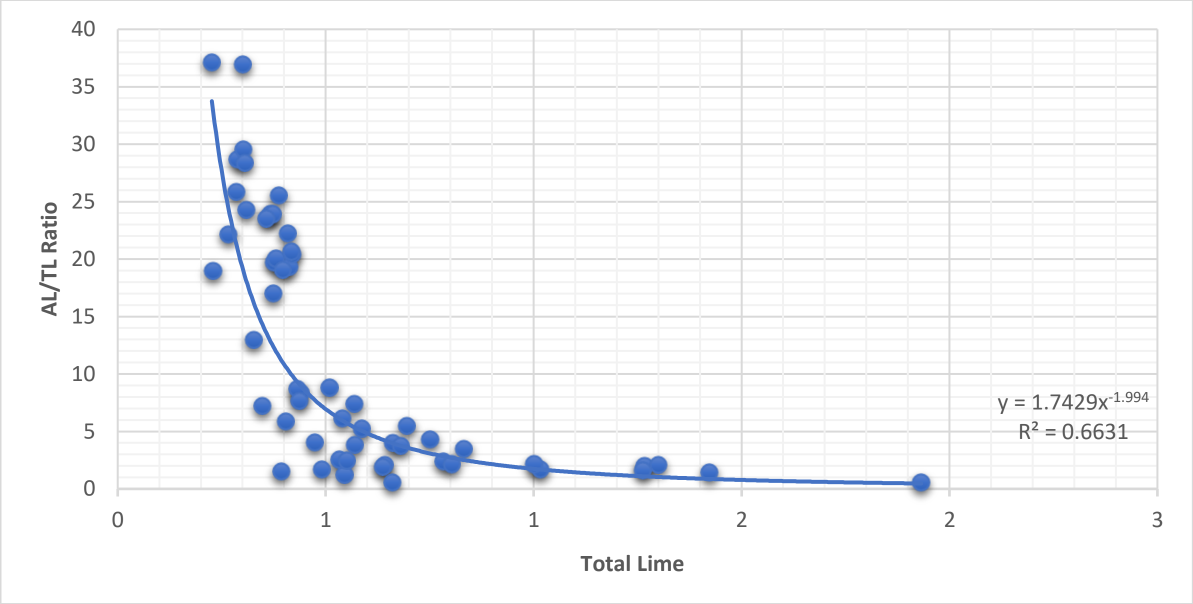

Figure 2. Relationships between total lime and active/total lime ratio (AL/TL Ratio)

The relationships between active lime and the active/total lime ratio are depicted in Figure 3. Similar to total lime, a negative relation was observed between the active lime/total lime ratio and active lime. As can be seen in Figure 3, AL/TL ratio decreases exponentially as the amount of active lime increases. Figures 2 and 3 indicate that deviations in the ratio are observed at low values of both total and active lime. This situation is thought to be associated with the measurement of relatively higher active lime contents, as explained earlier, when calcium on the exchangeable surfaces passes into solution, especially in conditions of low total lime. Therefore, this situation raises questions about the reliability of active lime analyses in low total lime contents. Obtaining more reliable results occurs with the increase in total lime amounts, eliminating this relative effect. On the other hand, the variation in the amount of surface areas associated with soil texture and particle size is also a factor influencing carbonate reactivity (De La Torre et al., 2010; Loeppart and Suarez, 1996).

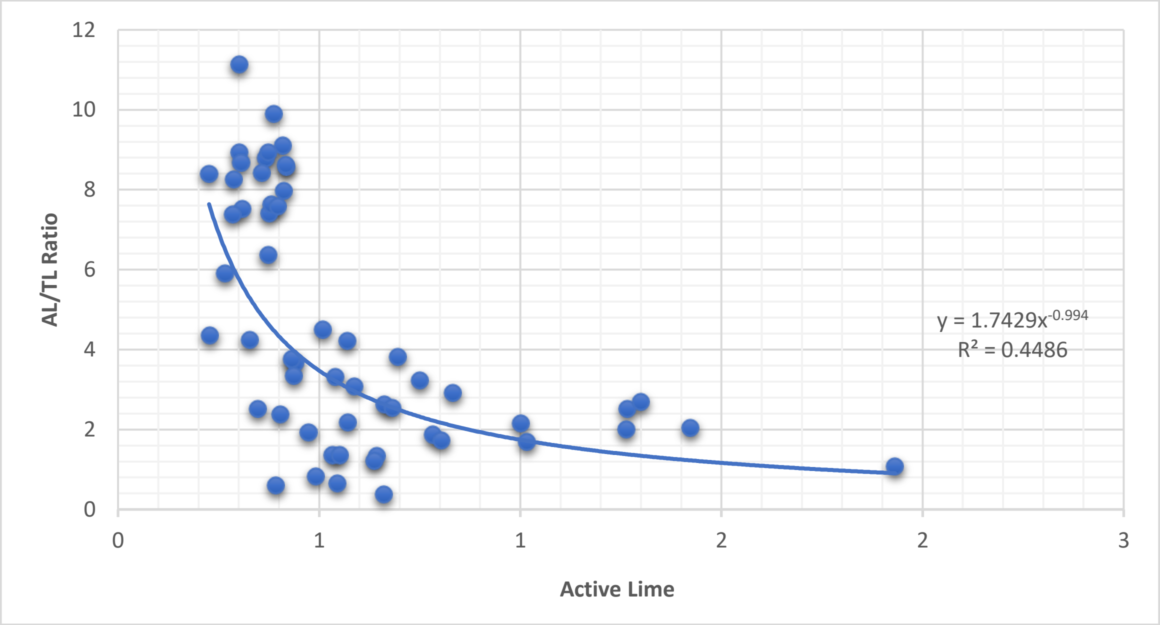

Figure 3. Relationships between active lime and active/total lime ratio (AL/TL Ratio)

Relationships among soil parameters

When examining the correlation values between soil parameters, a significant positive relationship at the 1% level was obtained between active lime and total lime (r=0.95**). Similarly, Ryan et al. (1981) emphasized a highly significant correlation between active and total lime. Carter (1981), on the other hand, found a moderately significant relationship between active lime and total lime (r=0.57**). As the total amount of lime increased, the proportion of active lime within the total was decreased (r = -0.63). A significant negative correlation at the 1% significance level was also observed between the ratio of active lime to total lime and active lime (r = -0.54). In other words, as the active calcium increases, its proportion within the total calcium decreases. In situations where total lime is low, the relative measurement of higher active lime contents can be explained by the exposure of exchangeable calcium. However, in cases of high total lime values, this effect disappears.

When analyzing the impact of active lime on the availability of metal micronutrients, a noteworthy inverse correlation at the 1% significance level was observed between active lime and DTPA-Fe concentrations (r=-0.48**). Şendemirci et al. (2016) similarly reported that the increase in total lime and active lime in soils is associated with a decrease in Fe availability. While Eyüpoğlu et al. (1999) indicated significant iron deficiencies in areas with high lime content, Cox and Kamprath (1972) emphasized that total lime alone is not sufficient for detecting iron deficiencies in soils. Additionally, Loue (1986) emphasized the necessity of determining active lime as a measure of iron availability in soils. No significant relationship was found between active lime and DTPA-Zn concentrations. Although Fahad (1988) stated that most of the applied Zn in calcareous soils can be retained by lime, Navrot and Ravikovitch (1969) reported that the particle size of lime is important for Zn retention. Therefore, the particle size of lime is also important in the size of active particles (<2 µm). A substantial positive correlation was observed between active lime and DTPA-Cu at a significance level of 5% (r=0.31*). In general, various positive or negative associations can arise between DTPA-Cu and pH and soil lime contents. A significant negative relation was found between active lime and DTPA-Mn at a significance level of 1% (r=-0.51**).

A statistically significant positive correlation at the 1% level of significance was observed between the total amount of lime and the pH value (r = 0.65**), as predicted. Likewise, a significant positive correlation at the 1% level of significance was discovered between the amount of active lime and the pH value (r = 0.67**). The negative correlation at the level of 5% was determined between total lime and EC (r= -0.30*). Abdulwahhab and Şeker (2020) also found that lime applications significantly decreased EC values in clayey soils.

A positive relationship at the 5% significance level was found between total lime and DTPA-Cu (r = 0.29*), while negative relationships at the 1% significance level were identified between total lime and both DTPA-Fe and DTPA-Mn (r = -0.40**, r = -0.50**, respectively). On the other hand, no significant relationship was found between total lime and DTPA-Zn. According to Machold (1968), solutions of calcareous soils usually contain a high proportion of Ca, and Ca and Fe ions compete for the same binding sites of clayey compounds, which reduces the availability of iron to plants.

When examining the relationship between soil pH and available Fe and Mn, a negative correlation at the 1% significance level was observed (r = -0.35**, r = -0.51**, respectively), and a positive correlation at the 1% significance level was identified between soil pH and available Cu (r = 0.44**). However, no significant relationship was found between soil pH and available Zn. Şendemirci et al. (2016) reported that as the total and active lime amounts increased in soils, the available Fe levels decreased. Alcántara et al. (2000) and De La Torre et al. (2010) emphasized the relationship between iron and active lime. According to Karaman (2012), the available manganese in soils increases with the decrease in soil pH, and a significant portion of it becomes reduced to toxic levels depending on the intensity of this decrease. However, manganese availability decreases with the increase in lime. Fitts and Nelson (1956) also emphasized the importance of considering soil pH as a significant criterion for deficiencies or excesses of micronutrients. They reported that, as soil pH moves from neutral to alkaline, deficiencies in micronutrients can be more common.

Table 5. Correlation values between measured soil parameters
*= p<0.05, ** = p<0.01, ***= Active Lime/Total Lime Ratio

	Active Lime	**Total Lime**	**AL/TL Ratio**	**pH**	**EC**	**Fe**	**Zn**	**Cu**	**Mn**
Active Lime	1								
Total Lime	0.95^{**}	1							
AL/TL Ratio* **	-0.54^{**}	-0.63^{**}	1						
pH	0.67^{**}	0.65^{**}	-0.58^{**}	1					
EC	ns	-0.30^{*}	0.34^{*}	-0.27^{*}	1				
Fe	-0.48^{**}	-0.40^{**}	ns	-0.35^{**}	ns	1			
Zn	ns	ns	ns	ns	ns	ns	1		
Cu	0.31^{*}	0.29*	ns	0.44^{**}	ns	ns	ns	1	
Mn	-0.51^{**}	-0.50^{**}	ns	-0.51^{**}	ns	0.58^{**}	ns	ns	1

CONCLUSION

The findings indicated that the active lime method proved to be more efficient in showing the availability of DTPA-Fe in comparison to the total lime method. This implies that active lime could potentially hold greater significance than total lime when it comes to establishing Fe deficiency in soils. In this study, active lime did not provide a higher determinacy than total lime in explaining the availability of metal micronutrients other than iron. Furthermore, due to the very close relationship between active lime and total lime, sufficient data could not be obtained to consider or recommend active lime analysis as a routine soil analysis. It is thought that more conclusive results can be obtained in future studies by including plants in the analysis of the relationship between active lime and total lime.

Acknowledgements

This study was conceived by Hüseyin DİKİCİ, laboratory analyses were conducted by Hatice Sünbül and Ömer Faruk DEMİR, and the article was written by Ömer Faruk DEMİR and Hüseyin DİKİCİ.

¥ : This research includes the data of MSc. thesis of first author.

Disclosure Statement

No potential conflict of interest was reported by the authors.

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