Turkish Journal of Agriculture - Food Science and Technology, 6(1): 51-54, 2018

Turkish Journal of Agriculture - Food Science and Technology

Available online, ISSN: 2148-127X
www.agrifoodscience.com,
Turkish Science and Technology

Determination of Suitable Extraction Method for the Available Iron (Fe) Content of Calcareous Soils

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ARTICLE INFO

Research Article

Received 20 September 2017
Accepted 17 November 2017

Keywords:
Iron
Wheat
DTPA
Calcareous soil
Extraction method

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ABSTRACT

The aim of this research was to determine the most suitable extraction method for the available iron contents of calcareous soils in Trakya Region, Turkey. For this purpose ten calcareous soil samples were taken from research area and five extraction methods (Lindsay and Norvell, Wear and Evans, Olson, Soltanpur and Mehlich methods) were used and three biological indices (dry matter yield, Fe concentration, Fe uptake) were compared. The plant biological indices were determined with wheat (*Triticum aestivum* L.) plant grown under greenhouse conditions. At the end of the experiment, the highest correlation coefficients (r) were determined between the 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA, 0.005 M DTPA + 1 M NH_4HCO_3 methods and the biological indices. The correlation coefficients (r) for the 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA method and the three biological indices were 0.648**, 0.780** and 0.656** respectively. For the 0.005 M DTPA + 1 M NH_4HCO_3 method, these coefficients were determined 0.595**, 0.637** and 0.625**, respectively. Consequently, these extraction methods were suggested for the determination of the available Fe contents of the calcareous soils in Trakya Region, Turkey.

DOI: https://doi.org/10.24925/turjaf.v6i1.51-54.1561

Introduction

The trend to more intensive crop production with higher yields and heavier use of nitrogen (N), phosphorus (P) and potassium (K) fertilizers increases the need for Fe and other trace elements in agriculture. Soil analyses are helpful in for determining whether a soil can supply adequate amounts of Fe for optimal growth (Adiloğlu, 2006; Yinanç and Adiloğlu, 2017).

Iron deficiency is one of the most common trace element problems in the world nowadays. Iron deficiency is seen frequently in high pH, high lime, and insufficient organic matter content and sandy soils (Adiloğlu, 2006; Lindsay and Schwab, 1982). Available Fe is inadequate in about 26.87% of turkey's soils and 10.4% Trakya Region (Eyüpoğu et al., 1998; Adiloğlu, 2012).

Despite the fact that several Fe extraction methods have been developed none of them was suitable to be a standard method (Loeppert and Iskeep, 1996).

Lindsay and Norvell (1978) suggested DTPA (pH: 7.3) method for the determination of available Fe content with regards to neutral and alkaline soils.

The 0.001 M EDDHA method was suggested for the determination of available Fe content in the USA. Because, the highest correlation coefficient was determined between this method and biological indices (Johnson and Young, 1973).

Adiloğlu (2006), has used eight extraction methods for the determination of available iron contents in Brown Forest Soils in Turkey. 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA and 0.005 M DTPA + 1 M NH_4HCO_3 methods were recommended by Adiloğlu for the determination of available iron contents in Brown Forest Soils in Turkey.

A research was carried out as a suitable method of the determination of available iron content of North Greece Soils. DTPA, Mehlich 3, Soltanpour and Schwab methods were suggested for North Greece Soils (Chatzistathis et al., 2014).

Krzysztof et al. (2015) have used six chemical extraction methods for the determination of available iron contents in Polish Soils. Researchers have determined the highest statistical relationships Mehlich 3 and Yanai (0.2 M CH_3COOH + 0.25 M NH_4Cl + 0.005 M $C_8H_8O_7$ + 0.05 M HCl) methods. Consequently, these methods were recommended for Polish Soils by these researchers.

A research was carried out in order to find out the available iron contents and determine the most suitable extraction method of available iron contents of Oxsisols and Ultisols in Brazil. The highest correlation coefficient was determined 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA and Mehlich 3 methods. Therefore, these methods were recommended by Sobral et al. (2013), as the most

suitable methods for determination of available iron contents of Oxsisols and Ultisols in Brazil.

In this research, the most suitable method for the determination of available iron content in calcareous soils was investigated.

Materials and Methods

Soil samples were taken at 0- 20 cm depth from 10 different cultivated calcareous soils in Trakya Region (Jackson, 1967). Soil pH (Thomas, 1996), lime (Loeppert and Suarez, 1996), organic matter amount (Nelson and Sommers, 1996) and texture (Gee and Bauder, 1986) were determined for each soil sample.

Some physical and chemical properties of the soil samples are given in Table 1. According to Table 1, pH values of soil samples ranged from 7.43 to 8.02, $CaCO_3$ contents were between 5.69% and 17.23%, organic matter amounts were between 0.23% and 1.02%, texture of soils samples were (C), generally.

The available Fe contents of the soil samples were determined through five different chemical extraction methods. These methods are 0.005 M DTPA + 0.01M $CaCl_2$ + 0.1 M TEA (Lindsay and Norvell, 1978), 0.05 M HCl + 0.012 M H_2SO_4 (Wear and Evans, 1968), 1 M NH_4OAc (Olson, 1948), 0.005 M DTPA + 1 M NH_4OAc (Soltanpur, 1991) and 0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3 + 0.015 M NH_4F + 0.001 M EDTA (Mehlich, 1984). Some properties of these extraction methods are given in Table 2.

Table 1 Some physical and chemical properties of the soil samples

Soil No	pH (1: 2.5)	$CaCO_3$, %	Org. matter %	Clay %	Silt %	Sand %	Texture Class
1	7.66	12.20	0.52	38.42	27.66	33.92	CL
2	7.97	8.58	0.76	35.23	25.61	39.16	CL
3	7.85	5.69	0.23	40.42	15.78	40.80	C
4	8.02	13.27	0.78	42.45	30.07	27.08	C
5	7.62	17.23	0.67	38.85	12.10	49.05	SC
6	7.98	16.52	0.98	41.08	33.10	25.82	C
7	7.80	7.98	0.76	38.33	18.85	42.82	SC
8	7.65	9.52	0.53	44.32	28.05	27.63	C
9	7.52	6.67	1.02	43.28	24.30	32.42	C
10	7.43	7.86	0.87	47.60	21.72	35.68	C

Table 2 Chemical extraction methods were used for the determination of available Fe contents of the soil samples.

Methods	Soil – solution ratio	Shaking time	Reference
0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA	1: 2	120 minutes	(Lindsay and Norvell, 1978)
0.05 M HCl + 0.012 M H_2SO_4	1: 4	15 minutes	(Wear and Evans, 1968)
1 M NH_4OAc	1: 4	30 minutes	(Olson, 1948)
0.005 M DTPA + 1 M NH_4HCO_3	1: 2	15 minutes	(Soltanpur, 1991)
0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3 + 0.015 M NH_4F + 0.001 M EDTA	1: 10	5 minutes	(Mehlich, 1984)

A greenhouse experiment was designed in a randomized complete block replicated three times. Air dried 2.5 kg soil was filled into plastic pots. Rumeli variety wheat (*Triticum aestvium* L.) was used as a test plant because it is sensitive to Fe deficiency (Martens and Westermann, 1991). Each pot was fertilized with 150 mg kg^{-1} N (NH_4NO_3) and 100 mg kg^{-1} P_2O_5 (KH_2PO_4), according to average application rates of N and P_2O_5 to wheat in this region. Three different rates of Fe (Fe_0:0; Fe_1:15; and Fe_2:30 mg kg^{-1}) were applied to soils as $FeSO_4$ compound. Twenty plants were left in each pot after the germination. The water content of the pots was adjusted to 80% of field capacity during the experiment period. Wheat shoots were harvested after 60 days. Harvested shoots were washed once tap water and twice distilled water and dried at 65°C. Dry matter yields were determined (Kacar and İnal, 2010).

Dried and ground plant materials were digested using HNO_3 + $HClO_4$ and Fe content of plants were determined with ICP-OES (Kacar and İnal, 2010). Dry matter yield, Fe concentration and Fe uptake biological indices were used as biological method.

Correlation coefficients (r) were measured between available Fe content of the soils according to five different methods and biological indices (dry matter yield, Fe content and Fe uptake) of wheat plants. Significance of the correlation coefficients (r) was checked at the 1 and 5% levels (Düzgüneş et al., 1987).

Results and Discussion

The Effects of Increasing Iron Application Rates

Dry matter yield of the wheat plants was affected by the Fe application. The highest dry matter yield was obtained from the Fe_2 (30 mg kg^{-1}). On the other hand, Fe concentration and Fe uptake of the plants increased with increasing Fe application (Table 3). The reason of this result may be positive effect of iron application to high pH value and high lime contents of the soil samples.

According to Table 3, dry matter yield of plants were determined between 2.22 and 3.18 mg kg^{-1}, Fe concentration of plants were determined between 92 and 147 mg kg^{-1}, and uptake of iron were determined between 226 ile 433 µg pot^{-1}.

The effect of Fe application on the biological indices of the wheat plants was determined to be significant at 1 % level and the results obtained are in agreement with earlier reports (Başar and Özgümüş, 1999; Adiloğlu, 2006).

The Available Iron Contents of Soils

Five extraction methods were used for the determination of available Fe content of the soil samples (Table 4). Available Fe varied widely depending on the extraction method used, reasons for which could be pointed out as the type, concentration, pH, shaking time, soil solution ratio of the extraction solution and variability observed in the physical and chemical properties of the soils used.

As shown in Table 4, the highest available Fe content of soil samples were determined with 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA; 0.005 M DTPA + 1 M NH_4HCO_3 method in comparison to other extraction methods. On the other hand, the lowest available Fe content of soil samples were determined with 1 M NH_4Oac and 0.05 M HCl + 0.012 M H_2SO_4 methods. These results also show that higher available Fe was determined using methods with chelate + salt (0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA) method in comparison to the methods using salt (1 M NH_4OAc method) and acid (0.05 M HCl + 0.012 M H_2SO_4 method).

The acid and salt methods of HCl + H_2SO_4, $MgCl_2$ and NH_4OAc, which gave lowest available Fe, are not recommended for the determination of Fe content in neutral and alkaline soils. The use of chelate and chelate + salt methods are suggested in these types of soils (Adiloğlu, 2006).

The Relationships

The correlation coefficients (r) determined between chemical extraction methods and biological indices are given in Table 5. Significant correlation coefficients were observed between all chemical extraction methods, except 1 M NH_4OAc method and the biological indices (dry matter yield, Fe content and Fe uptake) at 1% and 5% levels (Table 5). According to Table 5, the highest correlation coefficients (r) were determined between 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA and 0.005 M DTPA + 1M NH_4HCO_3 methods and biological indices. These correlation coefficients (r) determined were 0.648**, 0.780** and 0.656** for 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 TEA method and 0.595**, 0.637** and 0.625**, for 0.005 M DTPA + 1M NH_4HCO_3 method, respectively. The lowest correlation coefficient (r) was determined with 1 M NH_4OAc method. These correlation coefficient were 0.310, 0.296 and 0.340* with biological indices, respectively.

According to the results the order of significance for the extraction methods are as follows: 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA > 0.005 M DTPA + 1 M NH_4HCO_3 > 0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3 + 0.015 M NH_4F + 0.001 M EDTA >0.05 M HCl + 0.012 M H_2SO_4 > 1 M NH_4OAc.

Table 3 The effect of iron application on biological indices of wheat plant*,**

Soil	Dry matter yield, g pot^{-1}			Fe concentration of plant, mg kg^{-1}			Uptake of Fe by shoots, µg pot^{-1}		
No	Fe_0	Fe_1	Fe_2	Fe_0	Fe_1	Fe_2	Fe_0	Fe_1	Fe_2
1	2.46 ^{a}	2.78 ^{b}	2.96 ^{c}	92 ^{a}	112 ^{b}	123 ^{c}	226 ^{a}	311 ^{b}	364 ^{c}
2	2.38 ^{a}	2.63 ^{b}	2.87 ^{c}	102 ^{a}	115 ^{b}	126 ^{c}	243 ^{a}	302 ^{b}	362 ^{c}
3	2.22 ^{a}	2.46 ^{b}	2.69 ^{c}	110 ^{a}	121 ^{b}	138 ^{c}	244 ^{a}	298 ^{b}	371 ^{c}
4	2.35 ^{a}	2.70 ^{b}	2.93 ^{c}	98 ^{a}	110 ^{b}	120 ^{c}	230 ^{a}	297 ^{b}	351 ^{c}
5	2.53 ^{a}	2.86 ^{b}	3.12 ^{c}	112 ^{a}	127 ^{b}	138 ^{c}	283 ^{a}	363 ^{b}	431 ^{c}
6	2.46 ^{a}	2.85 ^{b}	3.14 ^{c}	102 ^{a}	118 ^{b}	132 ^{c}	251 ^{a}	336 ^{b}	415 ^{c}
7	2.52 ^{a}	2.83 ^{b}	3.18 ^{c}	108 ^{a}	128 ^{b}	136 ^{c}	272 ^{a}	362 ^{b}	433 ^{c}
8	2.51 ^{a}	2.74 ^{b}	3.05 ^{c}	102 ^{a}	128 ^{b}	137 ^{c}	256 ^{a}	351 ^{b}	418 ^{c}
9	2.42 ^{a}	2.65 ^{b}	2.89 ^{c}	110 ^{a}	135 ^{b}	147 ^{c}	266 ^{a}	358 ^{b}	425 ^{c}
10	2.45 ^{a}	2.78 ^{b}	3.02 ^{c}	110 ^{a}	130 ^{b}	146 ^{c}	270 ^{a}	361 ^{b}	393 ^{c}

*: Values of three replication average, **: Each biological indices was evaluated individually.

Table 4 Iron content in soils determined by different chemical extraction methods

Method	Available Fe content, mg kg^{-1}									
	1	2	3	4	5	6	7	8	9	10
0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA	4.78	5.52	3.24	7.86	4.45	6.53	2.12	5.07	3.19	6.32
0.05 M HCl + 0.012 M H_2SO_4	2.30	2.24	1.15	3.47	1.08	4.74	1.16	2.61	1.87	2.42
1 M NH_4OAc	2.12	1.89	1.02	3.41	2.16	2.87	0.56	3.21	1.16	2.45
0.005 M DTPA + 1 M NH_4HCO_3	4.42	4.89	2.76	5.41	3.20	4.98	1.46	4.24	2.62	5.27
0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3 + 0.015 M NH_4F + 0.001 M EDTA	3.46	2.96	4.21	6.85	4.27	4.87	1.02	3.64	3.04	5.12

Table 5 The correlation coefficients (r) were between chemical extraction methods and biological indices

Chemical extraction methods	Non application of Fe in pots (Fe_0)		
	Dry matter yield	Fe concentration of plant	Uptake of Fe amount from soil
0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA	0.648**	0.780**	0.656**
0.05 M HCl + 0.012 M H_2SO_4	0.378*	0.421*	0.385*
1 M NH_4OAc	0.310	0.296	0.340*
0.005 M DTPA + 1 M NH_4HCO_3	0.595**	0.637**	0.625**
0.2 M CH_3COOH + 0.25 M NH_4NO_3 + 0.013 M HNO_3 + 0.015 M NH_4F + 0.001 M EDTA	0.578**	0.460**	0.526**

*: P<0.05 **: P<0.01

Conclusion

According to the this research results, 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA and 0.005 M DTPA + 1 M NH_4HCO_3 methods, can be used confidently to determine the available Fe content of the calcareous soils of Trakya region because the highest correlation coefficients (r) were determined when these methods were used (Table 5). These methods were also suggested for various regional soils (Aydemir, 1981; Haddad and Evans, 1993; Adiloğlu, 2006).

Consequently all of the following methods i.e. 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA and 0.005 M DTPA + 1 M NH_4HCO_3 can be recommended in the determination of available Fe content of Trakya region calcareous soils because of the highest correlation coefficients (r) determined. On the other hand, these methods are suitable to certain physical and chemical properties of calcareous soils in this region. Results obtained can be applied to calcareous soils for available Fe content.

Acknowledgement

The abstract of this paper has been presented at the national conference "Turkey Natural Nutrition and Healthy Living 2015".

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