Mysore J. Agric. Sci., 52 (2) : 416-422, 2018

Evaluation of Extractants for Determination of Available and Estimation of Critical Limits of Zinc in Tomato Grown Soils of Eastern Dry Zone, Karnataka

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Abstract

Pot experiment was conducted in the green house of the Department of Soil Science and Agricultural Chemistry, UAS, Bengaluru, Karnataka, India during 2016-17 to determine available zinc and critical limits of zinc in tomato grown soils. The pot experiment consists of five treatments *i.e.*, 0, 5, 10, 15, 20 kg $ZnSO_4$ ha^{-1} with three replications and fifteen locations with completely randomized design with a total number of 225 pots. Among all the treatments Rec. NPK + $ZnSO_4$ @ 20 kg ha^{-1} recorded highest mean dry matter yields of tomato. The amount of zinc extracted by different extractants, were in the following order as Mehlich-3 - Zn > 0.1 N HCl - Zn > AB-DTPA - Zn > 0.01 N EDTA - Zn > DTPA - Zn > 0.01 M EDTA + 1 N NH_4OAc – Zn > 1 N NH_4OAc (pH 4.6) – Zn > 1 N NH_4OAc (pH 7) - Zn. Among the various extractants tried, DTPA - Zn gave positive and higher significant correlation with Brays per cent yield (r = 0.781). The next better extractants are Mehlich-3 (r = 0.726) and AB-DTPA (r = 0.576) which are also significantly positively correlated with Brays per cent yield. The critical limits of DTPA, Mehlich-3 and AB-DTPA in graphical methods are 1.12, 2.15 and 1.20 ppm while in statistical methods it is 1.16, 2.16 and 1.11 ppm, respectively. For tomato plant and fruit it is 34 and 66 ppm for graphical methods and 32 and 70.5 ppm for statistical method, respectively. The study indicated that tomato growing soils would respond to Zn application, when the soils contains less than 1.12 ppm DTPA extractable Zn.

Keywords : Available zinc, extractants, critical limits, mehlich-3, AB-DTPA, tomato

Soil and plant analysis is often used to determine the nutrient status of crops besides forming the guidelines for recommendations of the cost effective nutrient management practices. Extractants are chemical reagents used to extract nutrients from a soil which simulates the plant uptake. Numerous extractants have been developed by soil chemists to assess the relative nutrient status of soils and to serve as the basis for making nutrient recommendations. The extractants are designed to remove or extract a portion of soil available nutrient that can be correlated with some plant growth factors such as dry matter production, uptake and also quality parameters. The portion that is extracted represents only a very small fraction of the total amount of nutrient present in a soil and is related to the amount of the nutrient that may be potentially utilized by the plant over long run. The extractants used for a particular nutrient are normally well correlated with one another, but each extracting solution may extract different quantities of nutrients.

The fertilizer recommendations may not be relevant in the present day context, as there is appreciable decline in the organic matter level and fertility status of soil. Hence, several fertilizer recommendation approaches have been used based on soil tests to tackle the problem of NPK recommendations which were developed over 40 years ago by Agricultural universities in collaboration with ICAR and Developmental departments with respect to fertilizer application and to attain maximum yield per unit of fertilizer use which are being advocated to farmers and are included in the package of practices (POP). In this context, the concept of critical limits proposed by Cate and Nelson (1965) and Cate and Nelson (1971) is more appropriate for managing nutrient needs of different crops to reduce nutrient loss, besides preventing environmental pollution.

Critical limit is defined as the range of concentration of nutrient element at which growth of plant is restricted in comparison with that of plant at optimum nutrient level. The hypothesis is that the plant with nutrient concentration below the critical limit will respond to the addition of fertilizers and very low response observed above thecritical limit. The critical limit or level is quite often employed for wide variety of soils and crops, even though these critical limits may be different not only for soils, crop species but also for different varieties of given crop (Singh and Agarwal, 2007).

The importance of zinc has great thrust as it plays an important role in the production of chlorophyll, IAA, ascorbic acid and sugars. Among micronutrients, zinc deficiency is more predominant in soils of India and also in Karnataka. The general critical levels of zinc deficiency in Indian soils falls in the range of 0.6- 1.2 mg kg^{-1} by DTPA extractant (Rattan, 2017). For clear prediction of possible deficiencies these critical limits must be refined, as crops and soils vary widely in their nutrient supplying and utilization efficiency. However, such studies have not yet been carried out for zinc in tomato crop in Karnataka. Hence, the present study was conducted to determine the extracting efficiency of extractants and to establishing the critical limits for suitable extractants and for tomato crop.

Material and Methods

Fifteen surface soil samples from various locations (low, medium and high zinc soils) were collected from tomato growing soils of Eastern Dry Zone of Karnataka (Zone 5). The representative soil samples were air dried under shade and processed for further analysis for various physico-chemical properties and also for different extractants of zinc by adopting standard procedures. Pot experiments were conducted with 6 kg soil. The experiment consists of five treatments *i.e.*, 0, 5, 10, 15, 20 kg $ZnSO_4$ ha^{-1} along with RDF (250:250:250 kg N, $P_2O_5,$ K_2O ha^{-1}) with three replications and fifteen locations with completely randomized design with a total number of 225 pots (15 surface soils × 5 treatments × 3 replications). Seventeen days old seedlings were transplanted to the pots and irrigated to maintain field capacity. Proper plant protection measures were taken and the crop was harvested after 90 days. The tomato plant and fruit samples were washed with deionised water and then with distilled water. The samples were sundried and kept in paper bags and transferred to oven with controlled temperature of 60 °C. The oven dried samples were weighed for their dry matter yields and then ground to powder and stored for further analysis. Plant samples were digested by using di-acid mixture and analyzed by using AAS. Then Bray's per cent yields (BPY) and plant responses were calculated by using following formulae.

Brays per cent yield (BPY) = Control Yield / Maximum crop yield ×100

Crop response = Maximum yield – Control yield

The critical limits in soils and plants were determined by plotting BPY against soil extractants and separately with plant tissue zinc content, respectively following the method of Cate and Nelson (1965) and Cate and Nelson (1971).

Results and Discussion

Physico-chemical properties

The soils collected for pot culture experiments were sandy clay and sandy clay loam in texture with mean sand, silt and clay as 72.68, 7.93 and 19.34 per cent, respectively. The pH was slightly acidic to slightly alkaline (5.98 to 7.76) in nature. The EC ranges from 0.13 to 1.13 dS m^{-1} and low to high in organic carbon (0.42 to 1.81 %). The available nitrogen, phosphorus, potassium and sulphur ranged from 216.38 - 326.14 kg ha^{-1}, 43.72 - 124.50 kg ha^{-1}, 110.50 - 775.49 and 28.68 - 98.34 kg ha^{-1}, respectively. DTPA extractable Fe, Cu and Mn ranged from 1.90 - 14.06 mg kg^{-1}, 0.83 - 3.01 and 2.00 - 15.35 mg kg^{-1}, respectively. Iron ranged from low to high, where as Mn and Cu were high and sufficient in the studied experimental soils. Hot water boron content ranged from 0.42-1.19 mg kg^{-1}. The boron content ranged from low to high in the experimental soils.

Extractable zinc in soils by different extractants

The amount of zinc extracted by various extractants are presented in the Table I.

DTPA - Zn (mg kg^{-1})

The zinc extracted by DTPA ranges from 0.17- 4.89 with a mean of 1.67 and a median of 1.01 mg $kg^{-1}$with a standard deviation of 1.70. The zinc content of the experimental soils was categorized

TABLE I

Available zinc extracted by different extractants (mg kg^{-1})

Village	DTPA-Zn	Ammonium Bicarbonate - DTPA-Zn	Mehlich -3-Zn	0.1 *N* HCI-Zn	1 *N* NH_4OAc (pH 7)-Zn	1 *N* NH_4OAc (pH 4.6)-Zn	1 *N* EDTA Zn	0.01 *M* EDTA + 1 *N* NH_4OAc - Zn
Kanjanahalli-1	0.17	0.73	0.74	0.39	0.04	0.11	0.63	0.20
Kanjanahalli-2	0.26	0.56	0.94	0.56	0.16	0.27	0.57	0.34
Sadanahalli-1	0.26	1.02	0.87	0.90	0.09	0.36	0.84	0.90
Sadanahalli-2	0.19	0.90	0.69	0.49	0.09	0.23	0.75	0.41
Kethanahalli	0.24	1.21	1.81	2.13	0.10	0.55	1.82	1.29
Milapanahalli	0.70	1.96	2.17	2.74	0.15	0.77	1.44	1.67
Addegoppa	0.62	1.85	3.75	3.16	0.34	1.46	0.75	2.25
Yerranagenahalli	1.01	0.81	1.56	1.03	0.09	0.25	1.19	0.54
Madivala-1	1.17	0.54	1.26	0.80	0.07	0.25	2.86	0.31
Guddanahalli-1	1.16	3.51	2.15	4.29	0.41	2.36	3.69	2.90
Madivala-2	3.53	2.14	3.10	0.82	0.14	0.37	2.41	0.58
Garudanahalli-2	4.74	3.21	4.01	3.91	0.33	1.75	3.59	2.60
Vagari	2.82	2.98	3.42	4.45	0.93	2.76	3.83	2.65
Haripura-1	3.29	2.84	3.59	3.04	0.16	1.26	1.70	1.46
Haripura-2	4.89	3.59	5.42	4.05	0.31	1.87	1.61	2.45
Minimum	0.17	0.54	0.69	0.39	0.04	0.11	0.57	0.20
Maximum	4.89	3.59	5.42	4.45	0.93	2.76	3.83	2.90
Mean	1.67	1.85	2.36	2.18	0.23	0.98	1.85	1.37
Median	1.01	1.85	2.15	2.13	0.15	0.55	1.61	1.29
Standard Deviation	1.70	1.12	1.44	1.54	0.22	0.87	1.16	0.98

from low to high. DTPA extracted less amount of zinc than Mehlich-3, 0.1 *N*HCl, AB-DTPA and 0.01 *N* EDTA. Chelating agents reacting with zinc will form soluble complexes. These chelating agents will react with free zinc ion. During the reaction the chelated Zn accumulates in solution as the chelating agent combines with Zn^{+2}, causing more Zn to be released from labile solid phases. Soluble metal chelates are easily separated from solid matrix of the soils by filtration which can be measured by AAS. An advantage of chelate extractants over strong acids is that the pH of the extracting media can be carefully selected and controlled. This prevents the gross destruction of acid soluble soil minerals such as carbonates and oxides. These findings are in agreement with the reports of Manchanda *et al*. (2011).

Ammonium Bicarbonate-DTPA – Zn (mg kg^{-1})

The zinc extracted by Ammonium Bicarbonate - DTPA ranges from 0.54 - 3.59 with a mean and median of 1.85 mg kg^{-1} with a standard deviation of 1.12. AB-DTPA extracted more amount of zinc than DTPA and lesser than Mehlich-3 and 0.1 *N*HCl. This extractant was developed as a multi nutrient extractant in order to extract P, K, Ca, Mg, S and micronutrients which can be extracted with a single extraction process. Replenishment of free ion concentration in soil solution either through dissolution from the solid phases or desorption from the exchange phases in response to this complexation. The quantity of ions that accumulates during extraction reflects both the initial activity as well as the ability of the solid phases to replenish these ions in soil solution, thus simulating the root action. These findings were similar to that of the findings of Rattan (2017).

Mehlich-3 – Zn (mg kg^{-1})

The zinc extracted by Mehlich-3 ranges from 0.69 - 5.42 with a mean and median of 2.36 and 2.15 mg kg^{-1} with a standard deviation of 1.44. Mehlich-3 extracted higher amount of zinc when compared to all other extractants tried. The greater Zn extraction capacity of Mehlich-3, than the DTPA or AB-DTPA extractants might have been due to the presence of acid and EDTA in Mehlich-3. EDTA could be explained to its strong chelating agent capable of forming complexation and decreases adsorption of Zn on the clay particles and results in increasing the solubility of zinc in soils and dilute acids will partially dissolves metal oxides. These two mechanisms made Mehlich-3 to extract highest amount of zinc from the experimental soils. Similar reports were made by Takrattanasaran *et al*. (2010).

HCl – Zn (0.1 *N*) (mg kg^{-1})

The zinc extracted by HCl ranges from 0.39 - 4.45 with a mean and median of 2.18 and 2.13 mg kg^{-1} with a standard deviation of 1.54. 0.1 *N* Hydrochloric acid extracted more amount of zinc than DTPA and AB-DTPA and extracted less amount of zinc than Mehlich-3. Dilute acids remove Zn from soil solution, exchange sites on clays and soil organic matter and via partial dissolution of metal oxides. Their applicability is confined to most of the acid soils because they are not sufficiently buffered to extract meaningful levels of Zn from soils. These results are similar to that of the findings, reported by Manchanda *et al*. (2011).

NH_4OAc (pH 7) – Zn (1 *N*) (mg kg^{-1})

The zinc extracted by 1*N*NH_4OAc (pH 7) ranged between 0.04 - 0.93 with a mean and median of 0.23 and 0.15 mg kg^{-1} with a standard deviation of 0.22. It extracted lowest amount of zinc compared to all other extractants tried for the study. As 1*N*NH_4OAc (pH 7) is not a chelating agent and it has no acid component to displace the zinc from oxide bound minerals, it extracts very lower amounts of zinc than DTPA. These results are similar to that of the findings of Marchi *et al*.(2009).

NH_4OAc (pH 4.6) – Zn (1 *N*) (mg kg^{-1})

The zinc extracted by NH_4OAc (pH 4.6) ranges from 0.11 - 2.76 with a mean and median of 0.98 and 0.55 mg kg^{-1} with a standard deviation of 0.87. The amounts of zinc extracted by NH_4OAc remained low because in some high organic soils, ammonium acetate fails to extract better than DTPA. But values are higher when compared to 1*N* NH_4OAc (pH 7) due to its acidic pH. These results are similar to that of the findings of Marchi *et al*. (2009).

EDTA – Zn (0.01 *N*) (mg kg^{-1})

The zinc extracted by EDTA ranged between 0.57-3.83 with a mean and median value of 1.85 and 1.61 mg kg^{-1} with a standard deviation of 1.16. The highest concentration of available Zn extracted by EDTA than DTPA could be explained to its strong chelating agent capable of forming complexation and decreases adsorption of Zn on the clay particles and results in increasing the solubility of Zn^{2+} in soils. These results are in conformity with the findings of Bibiso *et al*. (2015).

EDTA (0.01 *M*) + 1 *N* NH_4OAc - Zn (mg kg^{-1})

The zinc extracted by EDTA 0.01*M* + 1 *N* NH_4OAc ranged between 0.20 - 2.90 with a mean and median of 1.37 and 1.29 mg kg^{-1} with a standard deviation of 0.98. There is a significant difference in the soil Zn extracted by DTPA and EDTA + 1 *N* NH_4OAc. The EDTA is a strong chelator of metals and is considered to act by chelating surface bound and solubilizing moderately soluble metal ions from the soil solid phase. The lower zinc extracted by this extractant is probably due to higher pH (8.6) of its solution with combination of neutral salt 1 *N* NH_4OAc, lower Zn was dissolved from the soil than that of DTPA solution with a pH of 7.3. These results are similar to that of the findings made by Zare *et al*. (2009).

At present, the chelating agents, if carefully selected, offers one of the most promising means of assessing the power of soils to supply the nutrients. Based on the amount of Zinc extracted by different extractants, the relative efficiency of the extractants were of the following order. Mehlich-3 - Zn > 0.1 *N* HCl - Zn > AB-DTPA - Zn > 0.01*N* EDTA - Zn > DTPA - Zn > 0.01 *M* EDTA + 1 *N* NH_4OAc – Zn >1 *N* NH_4OAc (pH 4.6) - Zn > 1 *N* NH_4OAc (pH 7) - Zn.

Correlations between different extractants of available zinc and with Bray's percent yield

A close relationship among different extractants suggested that the extractants tried for the study extracted varying quantities of available zinc but the degree of variation was closely related and the details

TABLE II

Correlation between extractants and brays per cent yield

	DTPA-Zn	AB DTPA - ZN	Mehlich -3-Zn	0.1 *N* HCI-Zn	1 *N* NH_4OAc (pH 7)-Zn	1 *N* NH_4OAc (pH 4.6)-Zn	0.1 *N* EDTA - Zn	0.01 *M* EDTA + 1 *N* NH_4OAc - Zn	Brays per cent Yield
DTPA-Zn	1.000								
AB-DTPA-Zn	0.768 **	1.000							
Mehlich-3-Zn	0.849 **	0.834 **	1.000						
0.1 *N* HCl-Zn	0.568 *	0.903 **	0.776 **	1.000					
1 *N* NH_4OAc (pH 7)-Zn	NS	0.636 *	NS	0.754 **	1.000				
1 *N* NH_4OAc (pH 4.6)-Zn	0.546 *	0.881 **	0.706 **	0.954 **	0.883 **	1.000			
0.01 *N* EDTA-Zn	0.537 *	0.628 *	NS	0.616 *	0.625 *	0.671 **	1.000		
0.01 *M* EDTA + 1 *N* NH_4OAc-Zn	NS	0.883 **	0.736 **	0.979 **	0.746 **	0.945 **	0.590 *	1.000	
Brays percent Yield	0.781 **	0.576 *	0.726 **	NS	NS	NS	NS	NS	1.000

** Significant at 0.01 level, * Significant at 0.05 level

are presented in Table II. DTPA was positively and significantly correlated with all other extractants except with 1 *N*NH_4OAc (pH 7) and 0.01 *M* EDTA + 1 *N* NH_4OAc. The correlation coefficient between DTPA and AB-DTPA is 0.768** and with DTPA and Mehlich-3 was 0.849**. The correlation between AB-DTPA and Mehlich-3 is 0.834**. With DTPA extractant, only Mehlich-3 and AB-DTPA extractants were significantly and positively correlated and remaining extractants were not much correlated as compared to AB-DTPA and Mehlich-3. Except 1 *N* NH_4OAc (pH 7) and all other extractants correlated significantly among themselves. This shows that these extractants can be tried to extract available zinc from the soils. All forms of extractable Zn were highly and significantly correlated with each other indicating that they could extract Zn from more or less similar pools from soil. These results are in conformity with the findings of Rahman *et al*. (2007); Zare *et al*. (2009); Muthukumararaja and Sriramachandrasekharan (2012).

Among the eight extractants, DTPA-Zn correlated highest with BPY followed by Mehlich-3 and AB-DTPA which gave better positive and significant relationship as compared to other extractants. The correlation coefficient between BPY and DTPA is 0.781** where as with Mehlich-3 and BPY is 0.726** and AB-DTPA with BPY is 0.576*. Hence, DTPA extractant is considered as best zinc extractant followed by Mehlich-3 and AB-DTPA extractant for influencing available zinc in soils. These results are similar to that of the reports made by Rahaman *et al*. (2007); Muthukumararaja and Sriramachandrasekharan (2012).

Critical limits of zinc for soils, tomato plant and fruit

Dry matter yields of tomato without zinc application varied from 43 to 72 g pot^{-1} in different soils having different levels of zinc comprising low, medium and high zinc status. The maximum yields worked out for different soils under different levels of zinc application was highest with the application of $ZnSO_4$ @ 20 kg ha^{-1}. Brays per cent yields worked out for tomato crop in different soils ranges from 55.56 to 93.24 g pot^{-1}. It was found to be highest in high zinc soils, whereas lowest relative yields was recorded in low zinc soils. The yield response was more in medium zinc and low zinc soils compared to that of high zinc soils (Table III).

TABLE III

Brays percent yields and yield responses of the experimental soils

Village	T_1	T_2	T_3	T_4	T_5	Check Yields (g pot^{-1})	Maximum Yields (g pot^{-1})	Brays Percent Yields	Yield Response
	Dry matter yields (g pot^{-1})								
Kanjanahalli-1	43	60	62	69	77	43	77	55.56	34.2
Kanjanahalli-2	49	50	55	66	68	49	68	71.62	19.3
Sadanahalli-1	54	63	67	72	70	54	72	75.00	18.0
Sadanahalli-2	50	56	62	68	74	50	74	67.24	24.2
Kethanahalli	54	66	64	68	74	54	74	73.61	19.5
Milapanahalli	66	64	80	82	98	66	98	67.35	32.0
Addegoppa	58	76	74	76	82	58	82	70.73	24.0
Yerranagenahalli	72	84	84	80	94	72	94	76.60	22.0
Madivala-1	60	72	90	80	94	60	94	63.83	34.0
Guddanahalli-1	62	62	80	96	90	62	96	64.58	34.0
Madivala-2	62	84	71	72	70	62	72	86.11	10.0
Garudanahalli-2	61	68	74	75	66	61	75	81.33	14.0
Vagari	62	68	70	72	72	62	72	85.56	10.5
Haripura-1	63	68	75	66	65	63	68	93.24	4.6
Haripura-2	70	72	78	74	72	70	78	89.74	8.0

T_1 Control ; T_2 Rec. NPK + $ZnSO_4$ @ 5 kg ha^{-1}; T_3 Rec. NPK + $ZnSO_4$ @ 10 kg ha^{-1}; T_4 Rec. NPK + $ZnSO_4$ @ 15 kg ha^{-1}; T_5 Rec. NPK + $ZnSO_4$ @ 20 kg ha^{-1}

The critical limits of DTPA extractable Zn for tomato was found to be 1.12 mg kg^{-1} and that of AB DTPA and Mehlich-3 extraction methods appeared to 1.20 and 2.15 mg kg^{-1}, respectively. The critical limits of Zn for DTPA, AB-DTPA and Mehlich-3 extraction methods by using statistical approach are 1.16, 1.11 and 2.16 mg kg^{-1}, respectively (Table II). Many researchers redefined critical limits of zinc for various crops and proposed critical limits which are slightly varied from crop to crop and location to location (Rahaman *et al*., 2007; Zare *et al*., 2009; Kausadikar *et al*., 2015 and Spalbar *et al*., 2017).

The critical limit of zinc in tomato plant and fruit in graphical method is 34 and 66 mg kg^{-1}, whereas in statistical method is 32 and 70.5 mg kg^{-1}, respectively. Comparison of both methods for estimating critical limits of the soil for tomato in the study showed that the results of two methods are close and are almost in equal range with slight deviation. The details are presented in the Table IV. Based on Cate and Nelson statistical method (1971) the range of the critical levels

TABLE IV

Critical limits of zinc (mg kg^{-1}) in soil, plant and fruit of tomato

Extractants	Graphical method	Statistical method	Percent variation between two methods
A)Critical limit of zinc in soil (mg kg^{-1})			
AB-DTPA Extractable Zn	1.20	1.11	3.57
DTPA Extractable Zn	1.12	1.16	7.50
Mehlich-3 Extractable Zn	2.15	2.16	0.46
B) Critical limit of zinc in plant (mg kg^{-1})			
Plant zinc	34	32	5.88
Fruit zinc	66	70.5	6.81

TABLE V

Range of critical levels (mg kg^{-1}) of zinc in soil, plant and fruit of tomato

Extractants	Low	Optimum	High
A)Critical limit of zinc in soil (mg kg^{-1})			
AB-DTPA Extractable Zn	< 0.96	0.97 - 1.53	> 1.53
DTPA Extractable Zn	< 1.08	1.08 - 1.99	> 1.99
Mehlich-3 Extractable Zn	< 1.98	1.98 - 2.64	> 2.64
B) Critical limit of zinc in plant (mg kg^{-1})			
Plant zinc	< 31.5	31.5 - 32.5	> 32.5
Fruit zinc	< 66.5	66.5 - 70.5	> 70.5

are established and are presented in the Table V. For categorization of zinc content for DTPA extractant the rating is < 1.08 is low, 1.08 – 1.99 is optimum and > 1.99 mg kg^{-1} is high. Similarly for AB-DTPA < 0.96, 0.97-1.53 and > 1.53 mg kg^{-1} are low, optimum and high and for Mehlich-3 the ratings are < 1.98, 1.98 – 2.64 and 2.64 mg kg^{-1} are considered as low, optimum and high. For tomato plant < 31.5, 31.5- 32.5 and > 32.5 mg kg^{-1}are considered as low, optimum and high. Similarly for fruit < 66.5, 66.5-70.5 and > 70.5 mg kg^{-1}are considered as low, optimum and high.

Among the eight extractants tried, DTPA-Zn correlated highest with Brays percent yield followed by Mehlich-3 and AB-DTPA which gave better positive and significant relationship when compared to other extractants.

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(*Received :* May, 2018 *Accepted :* June, 2018)