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RESEARCH ARTICLE

Critical Limits of Deficiency of Nickel in Intensively Cultivated Alluvial Soils

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Abstract
Although nickel (Ni) has been studied a lot as a pollutant, a very few studies have been conducted with this element as a plant nutrient. Present study was undertaken to evaluate the crop response of applied Ni and suitability of the chemical extractants for assessing the available Ni in soil using soybean as a test crop. Fifteen bulk surface (0–15 cm) soil samples with wide variation in physicochemical properties were collected from the cultivated fields of various locations. A greenhouse experiment was conducted to assess the response of soybean to applied Ni (0 and 5 mg kg^{-1}). There was 16.5 to 26.6% increase in the biomass yield of soybean to the applied Ni (5 mg kg^{-1}) over control. Effectiveness of diethylene triamine pentaacetic acid (DTPA) soil test for predicting the Ni content in plant improved, when the variation in soil pH was taken into account. Critical limit of deficiency of the DTPA-extractable Ni in soil was 0.17 mg kg^{-1}, and critical plant Ni concentration of deficiency for soybean was worked out as 0.20 mg kg^{-1}.

Keywords Nickel application · Crop response · Phytoavailability · Critical limit

1 Introduction

Nickel (Ni) was discovered as an essential plant nutrient in 1987 (Brown et al. 1987). However, its importance in plant nutrition has been realized long ago, after the discovery in 1975 that Ni is a component of the enzyme urease (Dixon et al. 1975). This Ni-dependent enzyme catalyzes the hydrolysis of urea to form ammonium ion (NH_4^+) and carbon di oxide (CO_2). Besides urease, it is also an important component of glyoxalases (Family-I), peptide deformylases, superoxide dis mutases (SODs), and hydrogenases (Chen et al. 2009; Nasibi et al. 2013). Eskew et al. (1983) reported that Ni-deficient soybean plant accumulates toxic levels of urea in its leaflet tips because of depression in urease activity in leaves. Similar disturbance in nitrogen (N) metabolism due to Ni deficiency has been reported in barley (Brown et al. 1990), wheat, ryegrass, sunflower, oil-seed rape, and zucchini (Gerenda!s and Sattelmacher 1997). Alibakhshi and Khoshgoftarmanesh (2015) reported that small amount of Ni can reduce the nitrate concentration of plants by increasing the activity of nitrate reductase (NR). Hydrogenase enzyme is responsible for the recycling of hydrogen produced during side reaction of nitrogenase in N-fixation process in legumes (Albrecht et al. 1979). There have been a few reports indicating acute Ni deficiency problems in the field crops, but it is likely that undiscovered (hidden) deficiencies may be critical in some areas (Alloway 2008). Positive response of crop to the applied Ni was reported in a limited number of studies (Brown et al. 1987). Mishra and Kar (1974) reported that seed treatment with Ni improved the shoot and root growth of wheat. In another study, Ahmad et al. (2009) reported that the low concentration (10 and 20 mg L^{-1}) of Ni significantly promoted seed germination and improved early seedling growth of sunflower (*Helianthus annuus* L.). Ojeda-Barrios et al. (2016) reported that application of the Ni exerted a positive effect on the nutritional state of foliar N, indicating a synergism between Ni and N. They also opined that Ni deficiency can have potentially harmful consequences for the metabolism and physiology of pecan trees. It is evident that whatever work has been carried out on response of crops to Ni application is mostly confined to the solution culture experiment. But from

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the crop production point of view, no systematic information is available even under intensive cropping on whether this element needs to be supplied through the external source.

Narrow range between deficiency and toxicity of micronutrients necessitates its precise application under the intensive cropping. Assessment of available micronutrients in soil is a prerequisite for its effective management in sustainable crop production (Yadegari, 2016). Although, a large number of extracting solution have been used to assess the plant available trace elements in soil, very few studies were carried out to assess the suitability of chemical extractant for assessing available Ni. Rahmatulla et al. (2001) used diethylene triamine pentaacetic acid (DTPA) soil test to assess the available Ni content in soils. On the other hand, 0.01 M strontium nitrate [$Sr(NO_3)_2$] have been used by a few workers to assess the available Ni in contaminated soils (Madden 1988; Kukier and Chaney 2004). However, no critical limit of deficiency of Ni was established in these studies. The importance of soil test as a diagnostic tool for predicting the response of crop to Ni application is determined largely by their ability to distinguish between soils deficient in available Ni and those are sufficient. This is accomplished by establishing the critical limit of deficiency in soil and plant. The suitability of extractant for available nutrients as well as their threshold values varies from soil to soil and crop to crop.

In India, virtually no systematic study has been conducted to establish and assess the critical limits of deficiency of Ni in agricultural soils, rates of accretion of Ni from the external sources, and responses of crops to applied Ni. Even no guide value is available under the Indian condition for assessing the phytoavailability of Ni in soil and plant. The present investigation was therefore undertaken to (i) evaluate the response of soybean to applied Ni in alluvial soil and (ii) assess the suitability of chemical extractants for determining available nickel in soil using soybean as a test crop. Since Ni plays very important role in N metabolism in legume crops, being a structural component of urease and hydrogenase, soybean has been chosen as a test crop. Alluvial soil was chosen for this study as this group of soil (e.g., Indo-Gangetic Plain) is reasonably fertile and contributes enormously toward India's food grain production.

2 Materials and Methods

2.1 Location of Soil Samples

In all, 200 surface (0–15 cm) soil samples (500 g each) were collected from the cultivated fields of Indian Agricultural Research Institute (IARI) farm, Keshopur, Madanpur, and Sonipat, which are located in and around National Capital Territory of Delhi. These soil samples were extracted with DTPA (Lindsay and Norvell 1978), and concentration of Ni in the extract was determined with the help of atomic absorption spectrophotometer (AAS). Out of these, 15 locations were selected to ensure the maximum variation in DTPA-extractable Ni content in soil (Table 1).

2.2 Characterization of Experimental Soil Samples

The soil samples were air dried, ground, and passed through a 2 mm sieve. Processed soil samples were analyzed for some selected soil properties using standard methods (Page et al. 1982). Mechanical composition of soil samples was determined by hydrometer method, while pH of soil samples was determined in 1:2 soil:water. Soil organic carbon (SOC) content in soil was determined by wet oxidation method. The cation exchange capacity (CEC) of the soil samples was determined by ammonium acetate method. Soil samples were extracted with citrate bicarbonate dithionite for free iron oxide (Fe_2O_3) and aluminum oxide (Al_2O_3) (Page et al. 1982). Iron content in the extract was determined with the help of AAS, and aluminum (Al) content was determined colorimetrically using Aluminon method. The soil samples were extracted for

Table 1 Location and description of experimental soils

Soil No.	Location	Description
1–4	IARI, New Delhi	Arable land under intensive cultivation with fresh irrigation water for more than seven decades
5–9	Keshopur, Delhi	Farmers' fields, which have been receiving sewage effluents for last 30 years under the Keshopur Effluent Irrigation Scheme, Delhi, India
10	Madanpur, Delhi	Agricultural lands of Madanpur, Delhi, which have been receiving irrigation either through tube well water or the river Yamuna
11	Madanpur, Delhi	Agricultural land has been irrigated with sewage effluents emanating from Okhla sewage treatment plant for last five decades
12–14	Madanpur, Delhi	Agricultural lands of Madanpur, Delhi, which have been receiving irrigation either through tube well water or the river Yamuna
15	Sonepat, Haryana	Fields (adjoining Atlas Cycle Factory) had been receiving industrial effluents for 15 years

Ni using 0.005 *M* DTPA (Lindsay and Norvell, 1978), 0.01 *M* $Sr(NO_3)_2$ (Madden, 1998), and 0.01 *M* calcium nitrate [$Ca(NO_3)$]. The Ni content in the extracts was determined by AAS.

2.3 Greenhouse Experiment

For the greenhouse pot experiment, 4 kg of soil was poured into plastic pots. A uniform basal dose (150% of recommended dose of NPK) of 20, 40, and 27 mg kg^{-1} of N, P_2O_5, and K_2O was added to the soil of each pot through urea, diammonium phosphate, and muriate of potash, respectively. Usually, in pot experiment, root density is considerably high as compared to that in field experiment. Hence, it is common practice to apply 150% of the recommended dose of NPK in a pot experiment (Barman et al. 2014b). Following the incorporation of these nutrients into soil, Ni was applied at the rate of 0 and 5 mg kg^{-1} as sulfate salt of Ni (Ni_2SO_4. $6H_2O$). All the NPK fertilizers and Ni were added in solution form and thoroughly mixed with soil. Each treatment combination was replicated twice in a completely randomized design using 60 pots. The soil in each pot was then irrigated to field capacity with deionized water and incubated for 1 week at ambient temperature. Fifteen soybean seeds (*Glycine max*; cv. PS 22) were sown, and after 2 weeks of sowing, a uniform plant population (six plants pot^{-1}) was maintained in each pot. The pots were watered daily. The plants were harvested at the flowering stage, i.e., after 55 days of sowing.

2.4 Soil and Plant Analysis

After harvesting, above ground parts of the plants were washed with deionized water and dried in a hot air oven at 60–70 °C. After attaining the constant weight, dry biomass yield was recorded. The dried plant samples were ground using mechanical grinder made up of stainless steel. Plant samples were then digested with di-acid mixture (HNO_3 and:$HClO_4$ in the ratio of 10:4) (Page et al. 1982), and Ni content in the digest was determined by the flame and graphite AAS depending on the concentration of Ni in the extracts.

2.5 Suitability of Chemical Extractants for Assessing Available Nickel

The ability of each extractant to determine the plant available Ni was assessed by examining the relationships between the amount of extracted Ni and each of Bray's yield and Ni concentration in plants grown in control pots (without applied Ni). Bray's yield of soybean was calculated as follows:

Bray's yield = (dry matter yield in control pot/dry matter yield at applied Ni in soil) × 100.

Hence, the maximum yield was used as denominator in the above mentioned formula for each soil other than the control.

The critical limits of deficiency of extractable Ni in soil and Ni content in plant were computed according to the statistical method of Cate and Nelson (1971). It is an iterative statistical procedure for separating the Bray's yield data into two classes based upon maximization of the class sum of squares in a one-way analysis of variance. The sum of squares reflects the weighted sum of squares of the differences between the Bray's yield means for the various classes and the grand mean. The formula is given below:

$$\text{Class sum of squares} = \left[\left(\text{Sum of Bray's yield for Class 1}\right)^2/\eta_1 + \left(\text{Sum of Bray's yield for Class 2}\right)^2/\eta_2\right] - \left[\left(\text{Sum of Bray's yields for both classes}\right)^2/\eta\right]$$

where η_1 = number of observations in Class 1,
η_2 = number of observations in Class 2, and
η = total number of observations ($\eta_1 + \eta_2$).

In other words, using this procedure, one can find out quantitatively the best divisions from the point of view of maximizing mean differences among the classes. Nutrient content in particular soil or plant sample is considered as a critical limit, which produces the maximum sum of squares.

2.6 Statistical Analysis

Simple correlation coefficients (r) of extractable Ni with Bray's yield and Ni content in plant (mg kg^{-1}) were worked to assess the suitability of extractants. Stepwise regression analyses were also carried out to evaluate the efficacy of different extractants in predicting Ni content in plant.

3 Results

3.1 Initial Properties of Experimental Soils

All the soil samples belong to Haplustept except one, i.e., soil sample no. 12, which is Usticpsamment. These soil samples were not representative of the study area as random sampling was done in order to ensure the wide variation in available Ni content in soil (1.39 ± 0.65 mg kg^{-1}) and other physical and chemical properties (Table 2). Overall, soil pH and EC were 7.79 ± 0.11 and 0.97 ± 0.26 dS m^{-1}, respectively. The CEC across the soils was 13.0 ± 1.03 (cmol p + kg^{-1}) with the clay content of 15.9 ± 2.39%, and the corresponding figure for SOC was 0.53 ± 0.08%. Free Fe and Al oxides were 0.30 ± 0.10 and 0.12 ± 0.06%, respectively. There was a wide variation in texture of experimental soils, which belong to five textural classes, viz., sandy loam, sandy clay loam, silt loam, sand, and loam.

Table 2 Physical and chemical properties of experimental soils

Location[$]	Mechanical composition			Textural class[#]	pH	EC (dS m^{-1})	CEC (cmol p + kg^{-1})	Organic carbon (%)	Free Fe_2O_3 (%)	Free Al_2O_3 (%)	DTPA-extractable Ni (mg kg^{-1})
	Clay (%)	Silt (%)	Sand (%)								
IARI, New Delhi (4)	26.5± 4.16	15.0± 2.65	58.5± 4.30	Sandy loam (1), Sandy clay loam (3)	8.07± 0.06	0.37± 0.13	13.7± 2.27	0.46± 0.07	0.32± 0.04	0.06± 0.004	0.31± 0.04
Keshopur, Delhi (5)	15.0± 1.81	24.3± 11.6	60.6± 12.3	Silt loam (1), sandy loam (4)	7.43± 0.23	1.74± 0.66	15.8± 1.38	0.74± 0.07	0.17± 0.01	0.04± 0.005	1.79± 0.30
Madanpur, Delhi (5)	7.62± 2.45	33.0± 13.2	59.3± 12.4	Silt loam (2), sandy loam (2), sand (1)	7.89± 0.16	0.76± 0.18	10.3± 1.72	0.37± 0.20	0.14± 0.03	0.24± 0.18	0.15± 0.04
Sonepat, Haryana (1)	18.8± 0.05	30.5± 0.50	50.6± 0.55	Loam (1)	8.03± 0.03	0.50± 0.005	12.2± 0.20	0.62± 0.02	1.64± 0.04	0.13± 0.005	9.91± 0.005
Overall (15)	15.9± 2.39	25.1± 5.76	58.9± 5.51	Sandy loam (7), silt loam (3), sandy clay loam (3) loam (1), sand (1)	7.79± 0.11	0.97± 0.26	13.0± 1.03	0.53± 0.08	0.30± 0.10	0.12± 0.06	1.39± 0.65

$Figures in parentheses indicate number of soil samples collected from respective locations

#Figures in parentheses indicate number of soil samples belong to respective textural classes

3.2 Extractable Nickel in Soil Samples

DTPA-extractable Ni content in soil (initial, i.e., without added Ni) varied from 0.06 to 9.91 mg kg^{-1}, whereas $Sr(NO_3)_2$- and $Ca(NO_3)_2$-extractable Ni in soil ranged from 0.43 to 58.5 and 0.41–171 μg kg^{-1}, respectively (Table 3). The mean value of DTPA-, $Sr(NO_3)_2$-, and $Ca(NO_3)_2$-extractable Ni in soil was recorded as 1.40 ± 2.49 mg kg^{-1}, 11.2 ± 16.8 μg kg^{-1}, and 16.6 ± 43.2 μg kg^{-1}, respectively.

3.3 General Appearance, Biomass Yield, and Nickel Content in Plants

Nickel deficiency symptoms include chlorosis and interveinal chlorosis in young leaves that progress gradually to necrosis. Other symptoms include poor seed germination and decreased crop yield (McCauley 2011). However, in the present study, no visual symptoms of deficiency appeared in soybean crop at control pots (grown without added Ni) throughout the growing period. Analysis of variance (ANOVA) related to main and interactive effects of soils and applied Ni on biomass yield of soybean at the flowering stage is presented in Table 4. Results indicate that biomass yield of soybean was affected by soils, which is significantly modified by the applied Ni in soil (interactive effect). Whereas, main effect of applied Ni was non-significant. There was a better growth of soybean plants grown on soil no. 12, 13, and 14 at 5 mg kg^{-1} of applied Ni. In these soil samples (12, 13, and 14), soybean crop responded positively to the applied Ni at 5 mg kg^{-1}. Biomass yield of soybean increased to the extent of 29, 20 and 20% over control in soil no. 12, 13 and 14, respectively. Whereas, mean Ni content insoybean plants ranged from 0.94-10.8 mg kg^{-1} across the soils (Table 5). Analysis of variance indicated that individual as well as interactive effects of soils and applied Ni on Ni content in soybean plant were significant. On an average, Ni content increased from 1.29 (control) to 3.90 mg kg^{-1} at 5 mg kg^{-1} of applied Ni in soil amounting to 26.1% increase over control.

3.4 Suitability of Extractants and Critical Limits of Deficiency of Nickel

Suitability of the extractants was evaluated by correlating the extractable Ni content in soil with the Bray's yield and Ni content in shoot of soybean separately (Table 3). DTPA-extractable Ni content in soil showed positive relationship with the Bray's yield (r = 0.60) and plant Ni content (r = 0.82), whereas $Sr(NO_3)_2$-extractable Ni in soil did not show any relationship, either with Bray's yield or Ni content in soybean plant. Calcium nitrate-extractable Ni contributed positively toward the Ni content in soybean plants, while it failed to show any significant relationship with the Bray's yield. An attempt has also been made to evaluate the efficacy of

Table 3 Nickel content in soil samples (without added nickel) and Bray's yield of soybean and their correlation coefficients (r) with plant availability parameters

Parameters	Range	Mean	Correlation coefficients (r)	
			Bray's yield (%)	Ni content in soybean (mg kg^{-1})
DTPA-extractable Ni (mg kg^{-1})	0.06–9.91	1.40 ± 2.49	0.60*	0.82**
$Sr(NO_3)_2$-extractable Ni (μg kg^{-1})	0.43–58.5	11.2 ± 16.8	0.49	0.48
$Ca(NO_3)_2$-extractable Ni (μg kg^{-1})	0.41–171	16.6 ± 43.2	0.50	0.74**
Bray's yield (%)	78.5–128	103 ± 13.0	–	–

* Significant at 5% probability level; ** Significant at 1% probability level

different extractants for soil Ni in predicting the Ni content in plant by taking the soil properties into consideration in the regression equation (Table 6). There was a significant improvement in the prediction coefficient (R^2) of regression equations based on both extractable Ni and pH as compared to that based on the extractable Ni only.

Critical limits of deficiency of Ni in soil and plant were worked out based on analysis of variance related to Bray's yield. While doing so, soils or plants were grouped into deficient (responsive) and adequate (non-responsive) categories, based on the maximum class sum of squares. Thus, critical limit of deficiency of DTPA-extractable Ni in soil and Ni content in plant (on dry weight basis at flowering stage) was worked out as 0.17 and 0.20 mg kg^{-1}, respectively for soybean (Tables 7 and 8).

4 Discussion

Wide variation in physical and chemical properties including the extractable Ni was observed across the experimental soils (Table 2). These variations are prerequisite to conduct pot experiment for establishing the critical limit of deficiency (Datta et al. 1994; 1998) because soil properties can affect

Table 4 Effect of nickel application on the biomass yield (g pot^{-1}) of soybean at flowering stage

Soil	Levels of applied Ni (mg kg^{-1})		Mean
	0	5	
1	10.2	9.9	10.0
2	14.2	14.1	14.2
3	14.8	14.0	14.4
4	11.8	11.8	11.8
5	16.2	16.0	16.1
6	13.0	12.3	12.6
7	13.5	12.5	13.0
8	15.7	12.3	14.0
9	12.7	12.0	12.3
10	10.5	10.2	10.3
11	16.6	15.1	15.8
12	10.9	13.8	12.4
13	11.1	13.1	12.1
14	12.1	14.1	13.1
15	9.67	7.94	8.81
Mean	12.9	12.6	
LSD ($P = 0.05$)	Soil = 1.58, Ni = NS, soil × Ni = 2.24		
ANOVA			
df	Soil = 14, Ni = 1, soil × Ni = 14, error = 30, total = 59		
Sum of squares	Soil = 230, Ni = 0.89, soil × Ni = 35.9, error = 3.7, total = 302		
Mean squares	Soil = 16.4, Ni = 0.89, soil × Ni = 2.57, error = 1.19		
F_{Cal}	Soil = 13.8 Ni = 0.75, soil × Ni = 2.16		

Table 5 Effect of nickel application on nickel content (mg kg^{-1}) in soybean

Soil	Levels of applied Ni (mg kg^{-1})		Mean
	0	5	
1	0.61	1.45	1.03
2	0.18	2.56	1.37
3	0.47	2.59	1.53
4	0.39	2.44	1.42
5	1.28	3.22	2.25
6	0.28	2.21	1.25
7	0.80	2.02	1.41
8	6.31	15.2	10.8
9	0.67	1.21	0.94
10	0.21	2.48	1.34
11	1.43	7.33	4.38
12	0.16	2.10	1.13
13	0.19	2.75	1.47
14	0.17	2.61	1.39
15	6.24	8.36	7.30
Mean	1.29	3.90	
LSD (P = 0.05)	Soil = 0.43, Ni = 0.16, soil × Ni = 0.64		
ANOVA			
df	Soil = 14, Ni = 1, soil × Ni = 14, error = 30, total = 59		
Sum of squares	Soil = 443, Ni = 102, soil × Ni = 61.8, error = 2.59, total = 610		
Mean squares	Soil = 31.7, Ni = 102, soil × Ni = 4.42, error = 0.09		
F_{Cal}	Soil = 366, Ni = 1179, soil × Ni = 51.1		

available micronutrients distribution, including soil organic matter, pH, moisture regime, etc. (Zhu et al. 2016). DTPA extracted much higher amount of Ni as compared to the other extractants, viz., $Sr(NO_3)_2$ and $Ca(NO_3)_2$. These results are in conformity with the findings of Kukier and Chaney (2004). In a fractionation study, Barman et al. (2015) reported the higher extractability of DTPA for Ni as compared to neutral salt solution. There was a significant variation in biomass yield across the soil, whereas the main effect of Ni was nonsignificant. However, the actual effect of the applied Ni, i.e., interactive effect of applied Ni and soil, was significant as evident in the case of soil no. 12, 13, and 14 (Table 4). Such results imply that the response of soybean to applied Ni is dependent on initial Ni status of soil. Nickel content in soybean plant was significantly modified with soil and external application of Ni irrespective of initial Ni status of soils, and Ni content increased due to the addition of Ni in the form of $NiSO_4$. Ojeda-Barrios et al. (2016) also reported the elevated level of Ni and other nutrients in foliage of pecan tree due to foliar Ni fertilization.

Table 6 Stepwise regression equations relating nickel content in soybean with extractable nickel and soil properties

Sl. No.	Regression equations	R^2	F
1	Y = 0.34 + 0.68 (DTPA-Ni)	0.67	26.8**
2	Y = 12.7 + 0.65 (DTPA-Ni) – 1.58 (pH)	0.79	22.2**
3	Y = 0.63 + 0.06 [$Sr(NO_3)_2$-Ni]	0.23	3.90
4	Y = 13.7 + 0.05 [$Sr(NO_3)_2$-Ni] + 1.77 (pH)	0.38	3.45
5	Y = 0.70 + 0.003 [$Ca(NO_3)_2$-Ni]	0.56	16.6**
6	Y = 17.9 + 0.004 [$Ca(NO_3)_2$-Ni] – 2.21 (pH)	0.78	21.8**

** Significant at 1% probability level; Y = Ni content in soybean (mg kg^{-1})

DTPA-extractable Ni content in these three soil samples, where positive response of Ni application was observed, ranged from 0.06 to 0.15 mg kg^{-1}. Soybean grown on soil no. 10 (DTPA-extractable Ni 0.12 mg kg^{-1}) did not respond to the Ni application even this soil contains similar amount of the DTPA-extractable Ni as soil no. 12, 13, and 14. Although such result is difficult to explain, this shows the differential ability of extraction/absorption of Ni by DTPA and plant roots in this particular soil. Positive response of soybean to applied Ni is attributed to the fact that it is an essential element for plants (Brown, 2007, Barman et al. 2014a). Eskew et al. (1983) reported that soybean plants grown under the low-Ni conditions in solution culture developed necrotic leaflet tips. A few other crops such as tomato, sunflower, maize, etc. responded positively to the low level of Ni in solution culture (Ahmad et al. 2009; Sabir et al. 2011). Earlier studies

Table 7 Analysis of variance method for computation of critical limit of deficiency of DTPA-extractable nickel in soil (mg kg^{-1}) for soybean

DTPA-extractable Ni (mg kg^{-1})	Bray's yield of soybean	Class sum of square	r^2
0.06	78.5		
0.12	103	330	0.14
0.12	84.4	741	0.31
0.15	85.5	1202	0.51*
0.20	103	1054	0.45*
0.31	110	747	0.32
0.37	100	792	0.34
0.38	106	706	0.30
0.39	101	792	0.34
1.39	106	757	0.32
1.50	106	756	0.32
1.58	101	1000	0.43
1.59	108	1114	0.47
2.98	128		
9.91	122		

Critical limit = (0.15 + 0.20)/2 = 0.175

*Critical limit is computed as the mean value of Ni content in soil having maximum value of R^2 and that in succeeding soil

indicated that soybean needs Ni supplementation for better urease activity, biological nitrogen fixation, increased organic acids, and improved protein synthesis (Macedo et al. 2016). Alibakshi and Khoshgoftarmanesh (2015) reported that small amount of Ni can reduce the nitrate concentration of plants by increasing the activity of nitrate reductase. The positive response of crops to Ni application could be due to stimulating effect of Ni on nitrogen metabolism (Seregin and Kozhevnikova 2006). It is involved in the activation of enzyme urease; hence, most Ni essentiality studies were focused on legumes due to higher urease activity in their seeds and transportation of absorbed nitrogen as ureides compounds within plant body (Sabir et al. 2011). As evident from the literature, very few studies were conducted to evaluate the response of crops to soil application of Ni. Such information particularly on soybean is virtually nonexistent.

Table 8 Analysis of variance method for computation of critical limit of deficiency of nickel content (mg kg^{-1}) in soybean

Ni content in soybean (mg kg^{-1})	Bray's yield of soybean	Class sum of square	r^2
0.16	78.5		
0.17	85.5	997	0.42
0.18	101	795	0.34
0.19	84.4	1310	0.56*
0.21	103	1134	0.48*
0.28	106	944	0.40
0.39	100	991	0.42
0.47	106	894	0.38
0.61	103	924	0.39
0.67	106	893	0.38
0.8	108	837	0.36
1.28	101	1102	0.47
1.43	110	1114	0.47
6.24	122		
6.31	128		

Critical limit = (0.19 + 0.21)/2 = 0.20

*Critical limit is computed as the mean value of Ni content in soybean plant having maximum value of R^2 and that in succeeding plant

Among the extractants, only DTPA-extractable Ni showed consistent positive relationship with the Bray's yield and Ni content in plant, while $Ca(NO_3)_2$-extractable Ni was significantly related with the Ni content. Chelating agent such as DTPA is capable of extracting dissolved metal in soil solution, metals held in sorbed and organically bound phases as well as somebound occluded metals in oxides and clay minerals. Whereas, neutral salt such as, $Ca(NO_3)_2$ and $Sr(NO_3)_2$ cannot extract metal from the more tightly bound pools such as specifically adsorbed, oxide bound and organically bound pools (Miller et al. 1986). These extractants extract Ni mainly from the water soluble and exchangeable pool. But extraction of Ni by the plant roots is not limited to these two pools only. In fact, loosely bound Ni is also accessible to plant roots besides water soluble and exchangeable. This mismatch is probably reflected in the poor relationships of $Ca(NO_3)_2$- and $Sr(NO_3)_2$-extractable Ni with Bray's yield and Ni content in plant. Besides extractable Ni in soil, pH is the most important soil property, which controls the transfer of Ni from soil to plants. Nickel availability in soil is generally reduced with the increase in pH, and same is true for the other cations (Macedo et al. 2016). Therefore, in the present study, an improvement was observed in predictability of soil test methods for assessing the available Ni.

Effectiveness of soil test lies in its ability to distinguish deficient soil in respect of particular nutrient from adequate ones. This key aspect of soil test is accomplished by establishing the critical limits in soil and plant. In the present investigation, critical limits of the deficiency of Ni in soybean plant and soil were 0.20 and 0.17 mg kg^{-1}, respectively. As Ni is very much essential for legume plants, there was a significant increase in plant biomass with the application of Ni in deficient soils under study. In respect to the critical limit of deficiency of Ni in soil (0.17 mg kg^{-1}), four soils (soil no. 12, 13, and 14) could be categorized as Ni deficient. Out of these four soils, three soils, i.e., 75%, responded to Ni application. This indicates that critical limit as established in the present study should be quite effective in distinguishing deficient soil from sufficient ones. Parida et al. (2003) obtained response of fenugreek to the external application of Ni on a sandy loam soil (Typic Ustochrept) with pH 7.8, EC 0.2 dS m^{-1}, SOC 0.56%,

and DTPA extractable Ni 0.3 mg kg^{-1}. Whereas, Rabie et al. (1992) did not obtain any response of faba bean, wheat, and sorghum on a soil having 0.7 mg kg^{-1} DTPA extractable Ni, 3.6% calcium carbonate and pH 8.0. Sabir et al. (2011) reported that maize responded positively to the Ni application having shoot concentration of 0.71 mg kg^{-1} in a solution culture. However, Narwal et al. (1991) obtained the positive response of corn (dry matter) to the Ni application in spite of corn containing 2.5 mg kg^{-1} of Ni on dry weight basis in control. Positive response in terms of chlorophyll content in pecan trees to foliar-applied Ni was earlier reported by (Ojeda-Barrios et al., 2016). It is clear that the critical limit of deficiency in soil and plant as worked out in the present study are relatively lower than those indicated in the earlier studies, although none of those experiments were designed to work out critical limits in soil and plants.

5 Conclusions

Soybean responded positively to the applied Ni at the rate of 5 mg kg^{-1}in light-textured soil samples. DTPA soil test can be used to assess the available Ni in soil. Inclusion of soil pH with DTPA-extractable Ni in soil further improved the usefulness of this soil test for available Ni. Critical limit of deficiency of the DTPA-extractable Ni in soil and Ni content in plant (on dry weight basis at flowering stage) was worked out as 0.17 and 0.20 mg kg^{-1}, respectively, using soybean as a test crop. Novelty of this paper is that the information particularly on the deficiency limit of Ni in soil and plant is first of its kind and will go a long way in helping the delineation of Ni-deficient soil.

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