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Zinc Soil Test Calibration Based on 0.1 N HCl Extractable Zinc and Cation Exchange Capacity from Upland Soils of Northern Nigeria

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Abstract: A soil-zinc calibration test study based on 0.1 N HCl extractable zinc and Cation Exchange Capacity (CEC) was carried out with upland soils of Northern Nigeria using maize (*Zea mays* L. moench) as a test crop. Treatments consisted of (i) no zinc and (ii) 10 mg kg⁻¹ Zn (greenhouse) and five levels of Zn as ZnSO₄ · 7 H₂O (field) replicated 3 and 6 times in a randomized complete block design respectively. Plant zinc concentration increased with levels of applied Zn. Higher zinc concentrations were observed in plants that received Zn application. Extractable Zn concentration increased with an increase in CEC. A positive correlation was also observed between extractable Zn and pH. HCl extractable-Zn correlated positively and significantly with Zn uptake in the first and second crops ($r = +0.735^{**}$ and $+0.575^{**}$), respectively. The amount of Zn extracted by 0.5 N HCl was also significantly correlated with CEC. The amount of Zn extracted by 0.1 N HCl was significantly correlated with the Zn uptake by the first and second crop. The regression equations obtained from this study indicated that the distribution of ions in the exchange site of soils should be considered in estimating plant Zn requirements in soils of the northern Nigeria. However available information on the soil CEC can be used to estimate or predict the Zn concentration of the plant tissue. Critical limits may not be required in estimating or interpreting plant Zn fertilization, as regression equations are more reliable and not empirical.

Key words: Zinc, CEC, Mehlich-1 extractable zinc, maize

INTRODUCTION

Calibration of any nutrient soil test can be used to predict the response of a crop to the nutrient. Such calibration study can be based on yield response to the applied nutrient. A perfect understanding of plant available zinc in soils (Yusuf *et al.*, 2005) its uptake and concentration in plant is desirable for calibration study (Junus and Cox, 1987).

Different extractants have been used to extract Zn from soils in an attempt to correlate the amount extracted with that taken up by plants, 0.1 N HCl seems to be the best extractant for Nigeria soils (Osiname *et al.*, 1973). This extractable zinc was found to positively correlate with cation exchange capacity (CEC). Mehlich 1 extractable zinc was also found to give a positive correlation with plant uptake (Wears and Evans, 1968) and a significant correlation with CEC ($r = 0.638$) in soils of the Nigerian savanna (Yusuf *et al.*, 2005).

Zinc reaction in soils involves mostly CEC (Junus and Cox, 1987), soil pH, clay content and organic matter (Loneragam, 1975). Lins and Cox (1988) observed strong correlation between soil pH and zinc availability. A positive correlation was also observed between 0.1 N HCl extractable Zn (HCl-Zn), CEC and total zinc content of the

soil in a study by Pam (1990). A reliable prediction equation may be developed for zinc requirement by correlating zinc uptake with CEC. Junus and Cox (1987) have shown that CEC may substitute for organic matter and soil clay content if determined by summation of exchangeable bases. Pam (1990) showed that the CEC of soils from the Nigerian savanna positively correlated ($r^2 = 0.263$) with organic matter and zinc uptake highly correlated with CEC. Since CEC is an important soil chemical property and easily determined in routine analyses, it seems logical to develop a model for soil zinc calibration using it.

The objective of this study was to calibrate extractable soil zinc with CEC in a zinc soil test and to examine the influence of this soil property on zinc availability.

MATERIALS AND METHODS

Greenhouse study: Thirty soils representing the soils of Southern, Northern, Sahel and Sudan savannas of northern Nigeria were collected for a greenhouse experiment (Table 1). The soils were mainly Alfisols and Entisols. The soil samples were air-dried, crushed and sieved through a 2 mm stainless steel sieve. The samples

were analyzed for pH (H₂O), organic matter was determined by wet oxidation (Walkley and Black, 1934) and available phosphorus was determined by Bray No. 1 method. The DTPA procedure of Lindsay and Norvell (1978) was adopted for determination of iron. The soils were analyzed for their initial extractable zinc by extraction with 0.1 N HCl (Wear and Sommer, 1948) soil test method. Total zinc content in both soil and plant samples was determined by Atomic Adsorption Spectrophotometer in a digest obtained with a mixture of HNO₃ and HClO₃ (5:1) adopting the method of Juo (1979).

The experiments were conducted on these soils using maize (*Zea mays* L.) variety TZSR-Y1 (yellow) as the assay crop. Two kilograms of the soils were weighed into plastic pots of 4 L capacity placed on a plastic receiver. A blanket application of N, P, K, S, Mg and Cu at the rates of 200, 100, 280, 120, 100 and 5 mg kg⁻¹, respectively as (NH₄)₂SO₄, NaH₂PO₄, KNO₃, (NH₄)₂SO₄, MgO and CuSO₄ · 5H₂O, respectively was applied to all soils from the 30 locations. These were applied in solution. The pots were irrigated with deionized water to field capacity. Treatments consisted of (I) no zinc (ZnO) and (ii) 10 mg kg⁻¹ Zn (Zn 10) supplied as ZnSO₄ · 7 H₂O and were replicated three times. Six seeds were sown in

each pot and thinned to three plants plot⁻¹, seven Days After Sowing (DAS). The plants (shoots and roots) were harvested six Weeks After Sowing (WAS) during each cropping, rinsed in distilled water, placed in large envelopes and dried in an oven at 65°C for 48 h. The oven-dried plant parts were weighed and ground. Total Zn contents in shoots and roots were determined after digestion, as described earlier.

Field calibration studies: The field calibration studies for the maize crop were conducted after the greenhouse studies at three sites. Samples from these sites had been used for the greenhouse study (Table 1). Five levels of Zinc (0, 2.5, 5.0, 7.5 and 10.0 kg Zn ha⁻¹) as ZnSO₄ · 7 H₂O were arranged in a randomized complete block design and replicated 6 times. Each plot received a basal application of 100 kg N, 22 kg P and 42 kg K ha⁻¹. The phosphorus, potassium and half of the nitrogen were applied just before planting and worked into the soil with a small hoe. The Zn treatments were applied two weeks after planting as side dressing.

Fifteen core samples were obtained at the 0-20 cm depth from the sites bulked together and mixed. The samples were air-dried and mixed through a 2 mm stainless steel sieve. The soil samples taken were subjected to the same analysis as described for the greenhouse study.

The fourth leaf from the top was sampled at 42 days after planting. Ear leaf samples were taken at 50% silking, leaf samples were rinsed with distilled water, enveloped and dried on an oven at 68°C for 48 h, ground, ashed and subjected to analyses as previously alluded to.

Statistical analysis: Simple and multiple regression analysis were used in selecting parameters to be included in the multiple regression prediction equations. GENSTAT (2003) was used to select the variables to be included in the multiple regression prediction equations. The variables used were squared terms and first-order interactions of 0.1 N HCl extractable Zn (HCl-Zn), CEC and plant Zn uptake (Zn_{upt}). Variables with only significant regression coefficient (F<0.05) were retained in the equation.

RESULTS AND DISCUSSION

Total and extractable Zn status of the soil is low with value ranging from 6 to 84 and 1.0 to 9.5 mg kg⁻¹ with mean 21.92±18.70 and 3.31±0.75, respectively (Table 2). This may be attributed to the parent material

Table 1: Location, history, taxonomy and ecological zone of the soils used

Location	History	Taxonomy	Ecological zone
Bakura	Cultivated	Entisol	Sudan Savanna
Bauchi	"	Alfisol	Northern Guinea Savanna
Bokkos	"	"	"
Dambatta	"	"	Sudan Savanna
Daura	"	Entisol	"
Dutsin-Ma	"	"	"
Gombe 1	"	Alfisol	Northern Guinea Savanna
Gombe 2	Fallow	"	"
Gummi North	Cultivated	"	Sudan Savanna
Gummi South	"	"	"
Gusau	"	"	"
Hadeija	"	Entisol	"
Hoss 1	"	Alfisol	Southern Guinea Savanna
Hoss 2	Fallow	"	"
Ikara	Cultivated	"	Northern Guinea Savanna
Kafin Maiyaki	"	"	Sudan Savanna
Kankiya	"	"	"
Katsina	"	Entisol	Sahel Savanna
Lafia	"	Alfisol	Southern Guinea Savanna
Maigana	"	"	Northern Guinea Savanna
Malumfashi 1	"	"	"
Malumfashi 2	Fallow	"	"
Ringin	Cultivated	Entisol	Sudan Savanna
Samaru 1	"	Ultisol	Northern Guinea Savanna
Samaru 2	"	"	"
Soba	"	Alfisol	"
Sokoto	"	Entisol	Sahel Savanna
Talatan Mafara	"	"	Sudan Savanna
Wudil	"	Alfisol	Sudan Savanna
Wurno	"	Entisol	Sahel Savanna

from which the soils are derived (Yusuf *et al.*, 2005). The mean 0.1 N HCl extractable zinc represents about 15% of the total zinc reserve ranging from 1.0 to 9.5 mg kg⁻¹ with a mean of 3.31 mg kg⁻¹. Plant zinc concentration however, increased with levels of applied Zn. Higher zinc concentrations were observed in plants that received Zn application. Zinc concentration in the first crop varied from 63.33 to 143.22 mg kg⁻¹ with an average of 89.62 mg kg⁻¹ while in the second crop, it varied between 92.08 and 229.17 mg kg⁻¹ with an average of 168.47 mg kg⁻¹. This wide variation may be due to differences in soil constituents and the rate of Zn fertilization (Yusuf *et al.*, 2005).

Extractable Zn concentration increased with an increase in Cation Exchange Capacity (CEC), indicating that quite substantial amount of Zn is being extracted from the colloidal surfaces. A positive correlation was also observed between extractable Zn and pH. Junus and Cox (1987) reported similar findings.

Response to Zn application to the first crop varied with Zn application from -0.80 to 0.63 g pot⁻¹, with a mean of 0.05 g pot⁻¹. Twelve soils representing 40% of the total from the southern Guinea to Sahel savanna did not respond to Zn application. Similarly, in the second crop, fourteen soils representing all the ecological zones did not respond to Zn fertilization. Zinc deficiency symptom was imminent in some of the soils as evident from interveinal chlorosis arising from the base of the leaf blades. Interestingly, most of these deficient soils responded positively to Zn fertilization.

Calibration and correlation: The soil critical level was estimated using Cate and Nelson (1971). The critical level of Zn in the soils was 2.20 mg kg⁻¹ when the first and second crops were considered. All soils with values below the critical value were Zn deficient, while those with values above the critical level were Zn sufficient (non-responsive).

HCl (0.5N) extractable-Zn correlated significantly with Zn uptake in the first and second crops ($r = +0.735^{**}$ and $+0.575^{**}$) respectively. The amount of Zn extracted by 0.5NHCl was also significantly correlated with CEC. Lombin (1983) observed similar relationship. An increasing CEC is an indication that essential nutrients

are increasingly available for good plant growth and development (Brady and Weil, 1999). Soils with higher CEC values are those with higher extractable Zn (Table 3). This is an indication that quite substantial amount of Zn is being extracted from the soil colloidal surfaces. The highly significant relationship between CEC and dry matter yields in the first crop ($R = +0.491^{**}$); second crop ($R = +0.367^{*}$) is an attestation to this. It is therefore logical to include CEC in a Zn soil test calibration.

Multiple step wise regression analysis was used to determine which soil property (ies) best predicts the amount of Zn extracted by 0.1 N HCl. Zinc uptake by the first (Zn_{upt1}) and second (Zn_{upt2}) crop as dependent variables entered the regression equation in order of decreasing contribution to the multiple correlations.

The amount of Zn extracted by 0.1 N HCl was significantly correlated with the Zn uptake by the first and second crop. When the selected combinations of CEC were substituted in the regression equations, higher correlation coefficients were observed in the plant Zn uptake in the first crop (Table 4). When the first-order interactions of the variables were included in the regression equation, the proportion of variability accounted for increased from 54 to 70% in the first crop and from 29 to 57% in the second crop, respectively.

Table 3: Total and extractable zinc and CEC of the soils used

Location	Total zinc (mg kg ⁻¹)	CEC (Cmol kg ⁻¹)	0.1 N HCl (mg kg ⁻¹)
Bakura	10.8	0.45	2.85
Bauchi	21.6	3.5	4.50
Bokkos	34.8	1.84	3.00
Dambatta	17.2	1.87	1.70
Daura	15.6	0.69	2.75
Dutsin-Ma	20.4	0.88	3.00
Gombe 1	13.2	0.46	1.45
Gombe 2	15.6	0.29	1.70
Gummi North	15.6	1.98	3.20
Gummi South	13.2	0.38	1.50
Gusau	10.4	1.65	3.25
Hadeija	16	1.80	3.75
Hoss 1	84	2.2	3.00
Hoss 2	68.42	2.7	2.65
Ikara	19.2	1.2	3.75
Kafin Maiyaki	12	2.55	3.15
Kankiya	19.2	2.27	3.75
Katsina	17.6	0.62	1.00
Lafa	25.2	1.50	3.25
Maigana	11.6	2.10	4.50
Malumfashi 1	8.4	1.63	3.60
Malumfashi 2	9.6	2.81	3.00
Ringin	12	1.17	2.50
Samaru 1	16.8	1.52	3.75
Samaru 2	13.2	1.20	3.50
Soba	18.8	2.92	9.50
Sokoto	11	6.12	5.00
Talatan Mafara	12	1.53	3.25
Wudil	8.4	1.77	4.00
Wurno	22.8	1.54	3.50

Table 2: Some chemical properties of the soils used

Soil parameter	Range	Mean*
pH (H ₂ O)	4.5-6.9	5.60
Organic matter (%)	0.28-2.13	0.78
Available P. (mg kg ⁻¹)	0.7-34.8	7.9
Total N (%)	0.01-0.11	0.05
CEC (cmol kg ⁻¹)	0.29-6.12	1.77
Extractable Fe (mg kg ⁻¹)	10.0-240.0	42.87
Total Zn (mg kg ⁻¹)	6.0-84.0	21.92
0.1 N HCl Zn (mg kg ⁻¹)	1.0-9.5	3.31

*Average of 30 samples

Table 4: Multiple regression equations relating plant zinc uptake to soil properties studied

$Zn_{upt1} = 50.8 + 6.37CEC - 14.57HCl-Zn$	$R^2 = 0.54$
$Zn_{upt1} = -10 + 37.99CEC - 8.51(CEC)(HCl-Zn) + 33.36 HCl-Zn$	$R^2 = 0.703$
$Zn_{upt1} = 2.8 + 24CEC + 31.22HCl-Zn - 3.5(CEC)^2 - 1.76(HCl-Zn)^2$	$R^2 = 0.66$
$Zn_{upt1} = -19.1 + 46CEC^2 + 36.11HCl-Zn + 3.98(CEC)^2 + 1.57(HCl-Zn)^2 - 16.27(CEC)(HCl-Zn)$	$R^2 = 0.69$
$Zn_{upt2} = 81.2 + 3.8CEC - 26.54HCl-Zn$	$R^2 = 0.29$
$Zn_{upt2} = -20.1 + 56.5CEC + 57.9HCl-Zn - 14.17(CEC)(HCl-Zn)$	$R^2 = 0.38$
$Zn_{upt2} = -41.5 + 18.6CEC + 83.3HCl-Zn - 5.56(HCl-Zn)^2 - 4.17(CEC)^2$	$R^2 = 0.42$
$Zn_{upt2} = 1.1 - 24.4CEC + 73.8HCl-Zn - 12.05(HCl-Zn)^2 - 18.7(CEC)^2 + 31.7(CEC)(HCl-Zn)$	$R^2 = 0.44$
Zn_{upt1} = Zinc uptake by the first crop; Zn_{upt2} = Zinc uptake by the second crop	

Similarly, the squared terms and the interactions of the variables increased the R^2 values to 0.69 and 0.44 in the first and second crops, respectively.

CONCLUSIONS

The regression equations obtained from this study indicated that the distribution of ions in the exchange site of soils should be considered in estimating plant Zn requirements in soils of the northern Nigeria. However available information on the soil CEC can be used to estimate or predict the Zn concentration of the plant tissue as observed by Junus and Cox (1987). Critical limits may not be required in estimating or interpreting plant Zn fertilization, as regression equations are more reliable and not empirical.

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