Efficiency of Zinc Ethylenediaminetetraacetate and Zinc Lignosulfonate Soluble and Coated Fertilizers for Maize in Calcareous Soil

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To test the effectiveness of coated and uncoated Zn fertilizers prepared with commercial zinc ethylenediaminetetraacetate (Zn−EDTA) and zinc lignosulfonate (Zn−LS), maize (*Zea mays* L.) was greenhouse cultivated in calcareous soils dosed with 5, 10, and 15 mg·kg^{-1} Zn. Crop yield increased from 25 g per pot in control to 42 and 39 g per pot when 15 mg·kg^{-1} Zn was added by applying Zn−EDTA-3 and Zn−LS-0 fertilizers, respectively. Although big amounts of Zn were taken by plants, concentrations in soil increased from 43 to 55 and 57 mg·kg^{-1} when 15 mg·kg^{-1} Zn as Zn−EDTA-3 and Zn−LS-3 was applied. Rosin coating improved the performance of Zn−EDTA fertilizers but scarcely did in fertilizers with Zn−LS. A large part of Zn applied remained in the soil in forms easily available to plants (water soluble plus exchangeable, organic complexed, and DTPA-extractable Zn), more so when the source of Zn was zinc lignosulfonate. Positive, significant correlations were obtained between the variables, yield, Zn concentration, and Zn uptake by the plant with respect to the available Zn and the first two sequentially extracted fractions (water soluble plus exchangeable and organically complexed). The Zn uptake by maize could be fairly accurately predicted from the sequential fractioning in the soil after harvesting using an equation obtained through multiple regression analysis.

Keywords: *Calcareous soil; maize response; slow release; soil Zn status; Zn−EDTA; zinc lignosulfonate*

INTRODUCTION

The deficiency of Zn has been frequently referred to maize, which is a crop very sensitive to it (Murphy and Walsh, 1983; Pal et al., 1989). In general this deficiency is not caused by the total concentrations in the soil of this element but by very low concentrations in the soil solution (Adriano, 1986). DTPA extraction is a method usually employed for diagnosing Zn deficiency (Liang and Karamanos, 1993); however the study of its distribution in soil fractions provides a better understanding of Zn behavior in relation to its availability for plant uptake (Adriano, 1986).

The lack of Zn constitutes an especially important problem in relation to crops grown in alkaline soils (Thind et al., 1990), where the pH and presence of active $CaCO_3$ induces the insolubilization of the cation, due to the adsorption of Zn by carbonates (Udo et al., 1970) and the precipitation of zinc hydroxide or carbonate (Singh and Sekhon, 1977), or the formation of insoluble calcium zincate (Sharpless et al., 1969). The presence of active $CaCO_3$ in the soil can also produce an excess of Ca absorption by the plant that hinders the Zn transport and uptake (Kabata-Pendias and Pendias, 1985). When fertilizers with $ZnSO_4$ are added to remedy the deficiency in soils of this type, reactions such as $ZnSO_4 + CaCO_3 \leftrightarrow ZnCO_3 + CaSO_4$ can be produced which decreases their efficiency. Other anionic species can also contribute to Zn insolubilization: sulfides should be mentioned due to the low solubility of their salts. For this reason chelate fertilizers are commonly used to overcome problems of oligoelement deficiency (Mortvedt and Giordano, 1969).

It has been shown that zinc ethylenediaminetetraacetate (Zn−EDTA) diffuses readily in calcareous soils and is even lixiviated (Modaihsh, 1990; Hernández et al., 1975). The utilization of less stable chelates such as zinc lignosulfonate (Zn−LS) (Murphy and Walsh, 1983; Loué, 1988), or of coatings in Zn−EDTA containing fertilizers causes a reduction in the lixiviated Zn, as demonstrated in soil column experiments (Alvarez, 1993).

To optimize the application of Zn as a deficiency corrector, Zn−EDTA or zinc lignosulfonate fertilizer was prepared and its physicochemical properties were studied (Rico et al., 1995). In this work these fertilizers were applied to a maize crop in order to investigate the plant response and the distribution and availability of the remaining Zn in the soil.

MATERIALS AND METHODS

Zinc Fertilizers. Characteristics and the preparation method of Zn fertilizers are described in a previous work (Rico et al., 1995). The composition of the coated and uncoated products used in this experiment is shown in Table 1. The coating material employed was a 1:1:1 mixture of natural, dismutated (Residis) and esterified (Resiester T) rosins together with tricalcium phospate. These materials are insoluble in water, and no chemical incompatability between them and the Zn sources employed has been observed.

Characteristics of the Experimental Soil. The soil used originated from Alcalá de Henares in the province of Madrid (Spain). Material for the experiments was taken from the ochric type surface A horizon (depth 0−25 cm). Samples were air-dried at room temperature (no losses of weight were observed at 110 °C) and sieved, and the fraction <2 mm was utilized. Some properties of the soil are texture = clay loam, pH = 8.5, organic matter (OM) = 1.3%, active $CaCO_3$ = 12.9%, cation exchange capacity (CEC) = 19.0 cmol·kg^{-1}, and soil classification is Calcixerollic Xerochrepts. Soil pH was measured in water at 1:2 (w/v) soil:water ratio, and OM was by potassium dichromate oxidation. Active $CaCO_3$ were measured with a Bernard calcimeter and CEC was assessed with

Table 1. Products Prepared with Zn−EDTA and Zn−LS on Urea and Coated with Rosin

product	Zn[a] (%)	N[a] (%)	coating[b] (%)
Zn−EDTA-0[c]	1.40	38.64	
Zn−EDTA-2	1.07	31.28	19.20
Zn−EDTA-3	0.96	24.38	36.10
Zn−LS-0	1.27	39.10	
Zn−LS-2	1.08	30.36	21.93
Zn−LS-3	0.89	26.22	32.77

[a] Coefficients of variation from 2 to 3%. [b] Coefficients of variation from 5 to 6%. [c] The number shows the respective order of each product in the series according to the total amount of coating.

both the NaOAc and NH_4OAc extraction procedures. All of the analysis procedures are described in the Official Methods manual of the Ministerio de Agricultura, Pesca y Alimentación (1994).

Zinc was fractioned in the soil as described by Murthy (1982) and by Mandal and Mandal (1986) with slight modifications. Fractions determined were F1, neutral 1 M NH_4OAc extractable (water soluble plus exchangeable); F2, 0.05 M $Cu(OAc)_2$ extractable (organic complexed); F3, 0.2 M $(NH_4)_2C_2O_4$, pH 3.0, extractable (amorphous sesquioxide bound); F4, citrate−dithionite buffered with $NaHCO_3$ extractable (crystalline sesquioxide bound); and F5, estimated by digesting the soil residue with 2:1 $HNO_3 + HClO_4$ (residual Zn). Total Zn was calculated as the sum of the five fractions extracted. The plant available Zn was assessed by extracting with diethylenetriaminepentaacetic acid (DTPA) (0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M triethanolamine, adjusted to pH 7.30), using Lindsay and Norvell's method (1978). Zinc was determined with an atomic absorption spectrometer (Perkin-Elmer, Model 3300).

Speciation of untreated soil used for the experiment gave the following Zn fractions in mg·kg^{-1}: F1, 0.08 (0.18%); F2, 0.77 (1.76%); F3, 0.47 (1.08%); F4, 1.28 (2.93%); and F5, 41.08 (94.05%). Total Zn expressed as the sum of fractions was 43.68 mg·kg^{-1}. DTPA-extractable Zn was 0.40 mg·kg^{-1} which represents 0.91% of total Zn; i.e., this soil was deficient in Zn for growing maize (Lindsay and Norvell, 1978; Loué, 1988).

Plant Studied in a Pot Experiment. Medium-long-cycle maize seeds of a variety extensively used as fodder were planted in pots with the selected soil. The maize was grown in 5 kg of dried soil placed in polyethylene pots with washed gravel in the bottom of the recipients to facilitate aeration and drainage. The nutritional condition of the soil for the maize crop was assessed using the EUF technique (Wiklicky and Nemeth, 1981), and taking this into account N, P, and K were supplied as a total base application in doses of 460, 200, and 200 mg·kg^{-1}, respectively. Amounts of Zn-containing fertilizer were added which gave 5, 10, and 15 mg·kg^{-1} soil weight basis. Tests were performed in triplicate.

Maize (*Zea mays* L.) used was the variety G. PAST (Funk's), hybrid 3 lines sweet No. 1. Seeds were germinated in a seed bed and were then transplanted into the prepared pots 12 days after sowing. Two seedlings were placed in each pot. The pots were taken to a greenhouse where the temperature varied between 18 and 40 °C and appropriate amounts of water were added to reach and maintain the field capacity moisture conditions with a limited drainage. Sixty days after transplanting—the end of the maximum plant growth period—the above ground part was cut, washed with tap water, and then rinsed with deionized water. The plant pieces were dried in a oven at 70 °C to a constant weight. Once weighed, they were ground and kept in sealed recipients for later analysis; each of these plant samples was subjected to wet digestion with an acid mixture (HNO_3−$HClO_4$, 1:1), and the Zn was determined in the resulting solutions.

RESULTS AND DISCUSSION

Two aspects were studied to discuss the behavior of the tested fertilizers: the effectiveness of their use on the crop and the status of the added Zn in the soil, which could be available to successive crops. The yield in dry matter and concentration of Zn in the plant was determined, and results are shown in Table 2. The quantities obtained when Zn was not added during fertilization were low, as expected from the amount of Zn extracted with DTPA in the soil, and lower than that obtained in an acid soil with similar available Zn due to the nature of the soil. The convenience of utilizing Zn in the fertilization was obvious since there were major increases with respect to the control sample in the two variables studied, even with the lowest dose of Zn added. The increase in the Zn dosage produced increases in dried matter and Zn concentration in plants that reached 1.7 and 3 times the value of the control, respectively. Those results are as good as those obtained with the same plant and fertilizers in an acid soil in which the increase in dry matter was a little smaller and the increase in Zn concentration a little bigger (Alvarez et al., 1996).

Table 2. Response of Maize to Graded Levels and Treatments with Zn−EDTA and Zn−LS in Soil

treatment	Zn applied (mg·kg^{-1})	dry matter (g per pot)	Zn concn (mg·kg^{-1})
control	0	25.09 ± 1.38[a]	36.70 ± 1.02[a]
Zn−EDTA-0	5	28.68 ± 1.54	65.25 ± 2.91
	10	39.45 ± 1.02	78.50 ± 3.05
	15	40.05 ± 2.47	90.30 ± 4.51
Zn−EDTA-2	5	29.26 ± 1.45	68.50 ± 2.15
	10	39.83 ± 2.01	91.75 ± 3.00
	15	41.10 ± 2.93	107.70 ± 5.24
Zn−EDTA-3	5	31.42 ± 2.11	68.75 ± 2.28
	10	39.81 ± 3.58	92.60 ± 1.51
	15	42.05 ± 1.65	110.10 ± 6.10
Zn−LS-0	5	27.70 ± 1.17	59.85 ± 3.68
	10	35.63 ± 2.36	75.25 ± 4.01
	15	39.26 ± 2.46	88.05 ± 3.04
Zn−LS-2	5	27.06 ± 2.97	59.00 ± 1.02
	10	35.20 ± 2.04	74.50 ± 4.07
	15	39.03 ± 2.81	86.50 ± 4.09
Zn−LS-3	5	27.11 ± 1.25	58.65 ± 3.22
	10	35.05 ± 2.41	73.00 ± 4.01
	15	39.17 ± 2.14	85.95 ± 3.14

[a] Averages for three replications (two plants each) with standard error.

Zinc concentrations over 50 mg·kg^{-1} were obtained with all treatments. This value is given as the minimum necessary for using this plant as ruminant feeding fodder (Périgaud, 1970). Zn plant concentrations increased with the three dosages applied but especially when Zn−EDTA was used as the source of the micronutrient, which agreed with the findings of Elgawhary et al. (1970), who indicated that EDTA increased the microelements diffusion in the plant. The differences between the amount of Zn uptake by plants as a function of both sources applied were larger, as was the product of dry matter and Zn concentration. A statistical study of these values was carried out, and significant differences were obtained between the applied dosages (significance level 0.01%) and between the fertilization treatments (significance level 0.02%).

The advantage taken by the plant of the amounts of Zn added as a function of the fertilizer used is shown in Figure 1. The conclusions suggested by the observation of that figure are supported by the results of the statistical analysis. The multifactor analysis of variance (confidence level 95%) for the percent utilization of applied Zn, with the fertilizer and dosage factors, indicated that significant differences existed between the fertilizers (significance level <0.01%) and between the dosage (significance level <0.1%). The multiple range analysis for the studied factors (fertilizer and Zn dosage), employing Duncan's separation of averages

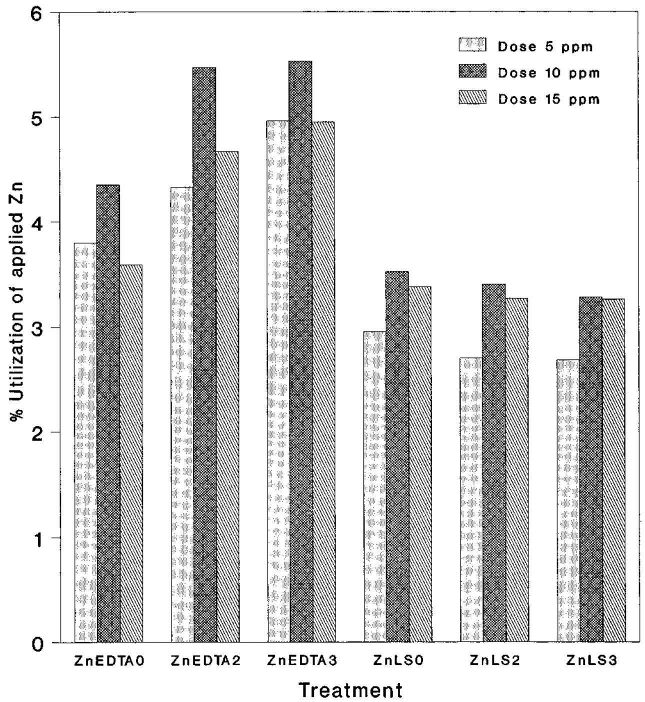

Figure 1. Effect of Zn−EDTA and Zn−LS application on the percent utilization of applied Zn in soil by maize plants.

method, indicated that no significant differences existed between the fertilizer treatments utilizing Zn−LS as the Zn source, but it established the existence of differences between those containing Zn−LS and those containing Zn−EDTA. For the treatments with Zn−EDTA differences between Zn−EDTA-0 and treatments with Zn−EDTA-2 and Zn−EDTA-3 were also demonstrated.

When the Zn source was Zn−EDTA, the percentage utilization was much higher with the three fertilizers tested and the coatings had favorable effects. With respect to the dosage Figure 1 shows that the best use was obtained with the 10 mg·kg^{-1} Zn dose in all cases, for which the analysis employing Duncan's method also demonstrated that the maximum of averages was established for this dosage. The variations observed were also higher when Zn−EDTA was used as the source of the microelement.

The values of Zn concentrations in different fractions and the DTPA-extractable Zn in the soil after cultivation presented in Table 3 showed that the amounts of Zn which remained in the soil and its chemical forms were dependent on the fertilization employed.

The increases produced in the total Zn concentrations in the soil (calculated as the sum of the fractions), which were noticeable in all cases, depended on the source used. The three fertilizers that contained Zn−LS behaved similarly, as happened with the Zn uptake by the plant. The Zn concentration in the soil with these treatments was higher than that produced with the Zn−EDTA fertilizers. In the latter case there was a higher concentration of the micronutrient in the soil with the most coated product (Zn−EDTA-3), which was also the one which led to higher Zn uptake by the plant. The greatest differences in behavior were between the treatments with Zn−EDTA-0 and the other fertilizers. When Zn−EDTA-0 was applied, the Zn extracted by plants and the remaining Zn in the soil were both the smallest. This can be explained due to the high stability of the Zn−EDTA complex, which diminishes the possibility of its being fixed by the soil. In the case, of Zn−EDTA-0, as there was no physical barrier, that is to say

Table 3. Zn Fractions in Soil with Different Doses of Zn−EDTA and Zn−LS Fertilizers after Maize Cutting (mg·kg^{-1})[a]

treatment	Zn applied	F1[b]	F2	F3	F4	F5	sum	DTPA Zn[c]
control	0	0.05	0.50	0.40	1.20	40.95	43.10	0.33
Zn−EDTA-0	5	0.20	2.61	0.60	1.22	42.00	45.63	2.02
	10	0.24	3.26	0.75	1.31	42.05	47.61	3.02
	15	0.26	4.08	0.95	1.44	42.85	49.58	4.03
Zn−EDTA-2	5	0.28	2.74	0.74	1.43	42.35	47.54	2.44
	10	0.32	3.91	0.95	1.65	43.20	50.03	3.75
	15	0.40	6.00	1.06	1.96	43.50	52.92	6.30
Zn−EDTA-3	5	0.38	2.77	0.78	1.62	42.35	47.90	2.50
	10	0.46	4.42	1.05	1.70	43.75	51.38	4.65
	15	0.64	7.25	1.08	2.05	44.80	55.82	8.61
Zn−LS-0	5	0.29	2.43	0.85	1.90	42.00	47.47	3.62
	10	0.36	4.60	1.96	2.16	43.35	52.43	5.41
	15	0.44	5.62	2.70	3.17	45.10	57.03	6.29
Zn−LS-2	5	0.29	2.36	0.88	1.62	42.45	47.60	3.02
	10	0.36	4.77	2.00	2.00	43.40	52.53	5.48
	15	0.40	5.88	2.34	3.05	45.90	57.57	6.67
Zn−LS-3	5	0.27	2.35	0.89	1.60	42.65	47.76	2.95
	10	0.36	4.70	1.97	1.98	43.90	52.91	4.89
	15	0.40	5.90	2.30	2.98	45.55	57.13	6.45

[a] Averages of three replications. [b] Coefficients of variation for all fractions (F1 to F5) from 2.9 to 8.1%. [c] Coefficients of variation for DTPA-extractable Zn from 2.5 to 4.8%.

coating, the chelate passed into the soil solution where it was partially leached by watering. Lixiviation diminishes with coatings and was practically inexistent for treatments with Zn−EDTA-3 (most coated fertilizer). The behavior related to leaching of these fertilizers was studied in soil columns in a previous work (Alvarez, 1993). Since Zn−LS was a less stable chelate and can decompose in the soil (Loué, 1988), the cation being fixed by the active components, the effect was that the migration produced by watering was minimal. On increasing the dosage applied, proportional increases in the Zn which remained in the soil were produced, which scarcely depend on the presence of the coating with this fertilizer. However, in treatments with Zn−EDTA-0 the percentage of Zn applied that remained in the soil decreased when the dosage was increased, this effect was lessened on using the controlled-release products (Zn−EDTA-2 and Zn−EDTA-3).

The amounts of Zn which remained in the fertilized soils, after harvesting the crop, were distributed in such a way that all the fractions were higher in comparison with the control soil, but the order of Zn concentrations in fractions F2 and F4 were inverted. The partition of the Zn was then the following: water soluble plus exchangeable < amorphous sesquioxides bound < crystalline sesquioxide bound < organic complexed < residual. Due to the characteristics of the soil employed the decomposition of the studied Zn chelates gave rise to the formation of insoluble compounds and the added microelement could be fixed, due to the high clay content of this soil since montmorillonite type smectites predominated in it.

With all the fertilization treatments the increases in the fractions were higher on increasing the dosage, the distribution between the different fractions depending on the source of Zn employed. The increase in fractions F3, F4 and F5 was higher when Zn−LS was used, corresponding to the highest total content achieved. However, this did not occur with fractions F1 and F2 with this source of micronutrient. These two fractions are very important with respect to the plant nutrition (Singh and Abrol, 1986). The small increments that were produced in F1 (whose maximum value cor-

Table 4. Linear Correlation Coefficients (*r*) between Zn in Maize and Different Forms of Zinc in the Soil (*n*=19)

soil Zn fraction	yield	Zn concn	Zn uptake
water soluble plus exchangeable	0.66[a]	0.78[c]	0.74[b]
organic complexed	0.85[c]	0.88[c]	0.87[c]
amorphous sesquioxide bound	0.46	0.34	0.35
crystalline sesquioxide bound	0.46	0.41	0.42
residual	0.70[b]	0.67[a]	0.68[a]
water soluble plus exchangeable plus organic complexed	0.84[c]	0.88[c]	0.87[c]
available	0.76[b]	0.80[c]	0.79[c]

[a]Significant at 0.5%. [b]Significant at 0.1%. [c]Significant at 0.01%.

responded to that of fertilizer Zn−EDTA-3) can be explained because this fraction contained the most labile form of Zn, and as such that which was most readily taken by the plant and lixiviated by watering. The increase produced in F2 (organic complexed Zn) reached a value 15 times higher than the control soil when 15 mg·kg^{-1} Zn−EDTA-3 was added.

The critical deficiency levels of available Zn in an alkaline soil, for a maize crop, established in the literature vary, but one of the highest is the value of 0.75 mg·kg^{-1} (extracted with DTPA−$CaCl_2$) reported by Thind et al. (1990). After harvesting the maize, for all of the fertilizers in this study, even at very low dosages, sufficient quantities of available Zn in the soil were left for a new crop. Even in the worst case the amount was more than double that of the critical level (Table 3). The concentrations increased with all applied dosages for all treatments, producing the higher extracted quantity for the fertilizer Zn−EDTA-3 at a dosage of 15 mg·kg^{-1} Zn. Comparing the fertilizers, Zn−EDTA-0 and Zn−LS-0, higher values were observed in the product which contained Zn−LS. The use of coatings increased the values obtained with different dosages for the Zn−EDTA micronutrient source while they hardly varied with the Zn−LS source.

The modification in the percentage of DTPA-extractable Zn with respect to the total, with the different treatments was significant compared to that of the control soil without fertilizer which had only 0.77% Zn. The highest percentages were obtained when dosages of 15 mg·kg^{-1} Zn were employed with the treatments using Zn−EDTA-2 and Zn−EDTA-3, obtaining values of 11.9% and 15.4%, respectively.

To establish relationships between the studied variables in plants and the extractions from the soils after harvesting, a simple linear regression analysis was carried out utilizing the data obtained with all of the fertilizer treatments and the control (Table 4). Positive correlations with higher or lower levels of significance existed between the yield, Zn concentration in the plant, and Zn uptake with the extracted fractions, except with the Zn associated with sesquioxides (F3 and F4). The most positive and meaningful correlations were obtained with fractions F1 and F2 and the summation of both (most labile fractions), as well as with the DTPA-extractable Zn fraction.

In general, it can be stated that the information supplied by sequential Zn fractioning and available Zn in the soil corresponded with that provided by the maize plant analysis. To calculate milligrams of Zn uptake by the plant (Y) as a function of DTPA extracted, the following equation was obtained:

$$Y = 1.04 + 0.40[\mathrm{DTPA}] \qquad R^2 = 0.65 \qquad (1)$$

A relationship between Zn uptake by the plant and Zn fractions successively extracted in the soil was established using multiple regression analysis (confidence level 95%). The following regression equation was obtained:

$$Y = -5.73 - 1.92[\mathrm{F1}] + 0.76[\mathrm{F2}] - 0.77[\mathrm{F3}] - 0.12[\mathrm{F4}] + 0.17[\mathrm{F5}] \qquad R^2 = 0.84 \quad (2)$$

This supported the possibility of establishing the amount of Zn uptake per pot, based on the contributions made by the different sequential Zn extractions. Moreover, regression coefficient values (eq 2) indicated the greater proportional input of the F2 extracted with Cu(OAc)$_2$ (organically complexed fraction), which corresponded to the higher correlation coefficients shown in Table 4.

CONCLUSIONS

The two sources of fertilizer employed, Zn−EDTA and Zn−LS, gave rise to a good response by the maize in the studied soil. However, the increments of yield and concentration of Zn in the plant were higher when Zn−EDTA was added.

The levels of Zn found in the soil, along with the quantities taken by the plant in the treatments with fertilizers without coating indicated that Zn−LS did not lixiviate while Zn−EDTA did. The addition of coatings to these fertilizers positively affected their behavior when Zn−EDTA was employed as a source of the microelement, while they had no influence when Zn−LS was used.

The levels of available forms of Zn (water soluble plus exchangeable, organic complexed, and DTPA extractable) which remained in the soil after harvesting of the maize were adequate to supply a sufficient quantity of Zn to a new crop.

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