Residual Available Copper and Boron in Soil as Affected by Zinc sulfate and Boric acid in a Zinc and Boron Deficient Soil

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Abstract: Micronutrients such as copper (Cu) and boron (B) are needed in small amounts, and there are also likely to be residual effects for some years after their application. A field experiment with maize plant grown on Zn and B deficient soil was conducted to evaluate the effect of Zn and B interaction on the residual available Cu and B content in the soil during 2009 at Fars Province, Iran. Treatments including five levels of Zn (0, 8, 16 and 24 kg ha^{-1} and Zn foliar spray) and four levels of B (0, 3, and 6 kg ha^{-1} and B foliar spray) in a completely randomized block design were set up. The findings showed that the in all treatments, the residual available Cu and B in the soil increased compared to its initial levels (before culture). The main effect of Zn and B on the residual Cu was insignificant relative to the no Zn and B level. No treatments, showed a significant difference on the residual Cu in the soil as compared with the control and also the effect of Zn-B interaction on the residual Cu was insignificant. In most treatments, the residual B in the soil decreased compared to its initial level levels (before culture). The Zn-B interaction was significant on the residual available B content in the soil. The presence of Zn prevented from increase of the available B remaining in the soil by B use relative to the soil B content before culture. Application of a high amount of Zn in the soil decreased residual available B in the soil relative to the no Zn level.
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Introduction

Zinc deficiency is a very important nutrient problem in the world's soils. Total Zn concentration is in sufficient level in many agricultural areas, but available Zn concentration is in deficient level because of different soil and climatic conditions. Soil pH, lime content, organic matter amount, clay type and amount and the amount of applied P fertilizer affect the available Zn concentration in soil (Adiloglu, 2006). Zinc is an essential nutrient for all plant crops. Chemically, Zn has some similarities with Fe and Mn, and in plant uptake there can be competition between these elements (Neue et al., 1998). Furthermore, high levels of phosphate in soils can strongly reduce Zn availability (Marschner, 1995). As regards agriculture, according to Cakmak (2002) Zn deficiency is the most widespread soil micronutrient deficiency in the world. Availability of Zn for plants is particularly low in calcareous and alkaline soils, while absolute Zn contents tend to be low in highly weathered acid tropical soils. Almost half of the agricultural soils from India, one third of the agricultural soils in China, and 50 per cent of cultivated land in Turkey are considered Zn-deficient for plants (Frossard et al., 2000; Gupta, 2005). Other more location specific studies report on low soil Zn contents in, for example, Iran (Aref, 2010). In spite of the fact that the total amount of Zn in the soil is relatively high, but a small fraction of it is available to the plant. Numerous factors affect Zn availability, including the soil calcium carbonate content, which reduces the Zn availability in the soil (Mandal et al., 1992). Among the micronutrients, Zn deficiency is perhaps most extensive in the world. Zinc deficiency is most common in low- and high pH soils, low- and high organic matter, sandy, sodic, calcareous soils and waterlogged without ventilation (Takkar and Walker, 1993). Corn is among the plants most sensitive to Zn deficiency (Tandon, 1995).

Boron regulates transport of sugars through membranes, cell division, cell development, and auxin metabolism. Without adequate levels of B, plants may continue to grow and add new leaves but fail to produce fruits or seeds. A continuous supply of boron is important for adequate plant growth and optimum yields (Mahler, 2010). The total boron content of most agricultural soils ranges from 1 to 467 mg kg^{-1}, with an average content of 9 to 85 mg kg^{-1}. Available boron, measured by various extraction methods, in agricultural soils varies from 0.5 to 5 mg kg^{-1}. (Gupta, 2007). It appeared that the percentage of soils deficient in B varied from 0 to 69 percent, thus suggesting that multiple micronutrient deficiencies at more localized level might be much more common than based on only Zn, Fe, Cu and Mn (Nube, 2006). Boron deficiency is common in sandy and highly calcareous rich soils since there is an interaction between the Ca ion and the available B and high Ca levels at high pH reduces B uptake (Marschner, 1995). Soil texture, soil organic matter content, and soil moisture (annual precipitation, irrigation) are the three most important factors affecting boron availability in soils. Coarse textured soils (sands, loamy sands, sandy loams) that are low in organic matter are often low in plant-available boron. Boron deficiencies are especially pronounced in high rainfall areas (greater than 25 inches) where boron may have been leached from the soil profile. Over-irrigation may cause the same results. The availability of boron in the soil is also influenced by pH. Maximum boron availability occurs between soil pH 5 and 7 (Mahler, 2010). Copper is an essential element for plant growth. However, its presence in the soil in quantities lower or greater than the optimal amount could adversely affect plant growth (Tucker et al., 1995).

Soil fertility is an important factor, which determines the growth of plant. Soil fertility is determined by the presence or absence of nutrients i.e. macro and micronutrients. The availability of micronutrients is particularly sensitive to changes in soil environment. The factors that affect the contents of such micronutrients are organic matter, soil pH, lime content, sand, silt, and clay contents revealed from different research experiments. There is also correlation among the micronutrients contents and above-mentioned properties (Nazif et al. 2006). Iron, aluminum, and manganese oxides; organic matter; and phosphates, carbonates, and sulfides are important sinks for trace elements in soil–residual systems. The pH of the soil–residual system is often the most important chemical property governing trace element sorption, precipitation, solubility, and availability (Basta, 2005). In agriculture, micronutrients are an issue of increasing interest and concern. Many research activities are being undertaken which address the relationships between micronutrient provision to plants and associated crop growth, and trace elements such as Zn, Mn and Cu are increasingly recognized as essential when aiming for better yields (Mann et al., 2002; Bhadoria et al., 2003; Rashid and Ryan, 2004; Welch and Graham, 2004; Gupta, 2005; He et al., 2005).

The interaction among nutrient elements is very important for plant nutrition. Boron x Zn interaction among these interactions has been curicial in the Zn deficient soils, in recent years (Alkan et al., 1998). Copper, Zn, Mn and P are Fe antagonists, and high levels of these elements in soils (or in fertilizer) can reduce Fe uptake by plants. Thus, information on extractable Fe, for example using DTPA4, is generally of much more relevance than information on the absolute levels of Fe contents in soils (Nube, 2006). The negative effect of high P suggest an induced deficiency of another element, possibly Zn, Cu, Fe or Mn (or a combination of these micronutrients), as P is known to have an antagonistic effect on these micronutrients. (Nube, 2006).

In a study in the China the residual effect of 1.1 kg B/ha remained fully effective in correcting B deficiency in oilseed rape for 2 years in the Inceptisols, whereas the residual effect of 1.65 kg B/ha continued to correct B deficiency for at least 3 years in both the Inceptisols and the Ultisol (Yang et al., 2000). Aref (2007) by studying the effect of Zn and B on the residual nutrients in the soil observed that P, Fe, Mn and Zn increased relative to its initial levels. Yang et al., (2000) reported that foliar application of B fertilizer generally corrected B deficiency for oilseed rape but showed limited residual effect in the following years after application.

In agriculture, very little research considers the entire range of micronutrients that are essential for plants (Nube, 2006). The critical level of Zn in the soil in mg kg^{-1} by DTPA extraction, has been reported by Darajeh et al. (1991) for corn to be 0.8, by Agrawala (1992), 0.8, Sharma and Lai (1993), 0.6, Terhan and Gerval (1995), 0.75. Lindsay and Norwell (1978), using DTPA extraction introduced critical limits of the soil Zn content as low if less than 0.5 mg kg^{-1}, medium if between 0.5 to 1 mg kg^{-1} and sufficient if more than 1 mg kg^{-1}. Nijjar (1990) has reported the critical B level by the hot water method in calcareous soils as 0.5 mg kg^{-1}. The critical level of Zn in the soil by DTPA extraction, has been reported by Agrawala (1992), 0.78, and Sims and Johnson (1991), 0.1-2.5 mg kg^{-1}.

By measuring the residual nutrients in the soil after harvesting the crop, we can use many desired relations for better management in the culture. If the amount of the residual available element in the soil is at a high level, this represents less uptake by the plant or less fixation by soil particles and if it is at a low level, this represents more uptake by the plant or more fixation by soil particles. Of course, in addition to uptake and fixation, other factors such as uptake of elements from unavailable form to available form or vice versa as well contribute to the amount of residual available element in the soil. Therefore, the objective of the study was examination of the effect of Zn and B on the residual available Cu and B in the soil after harvesting corn so that we are able to plan for Cu and B use in the next cultures.

Materials and Methods

The field study was conducted at the farm of Aref in Abadeh Tashk, Fars province of Iran, on the corn (*Zea mays* L.), cultivar "Single Cross 401" during 2009 cropping season. The site is located 200 km northeast of the Shiraz, with latitude 29° 43' 44" N and longitude 53° 52' 07" E and 1580 m altitude. Composite surface soil samples (0-30 cm) were taken from the site before the experiment was initiated. This soil had a loam texture, pH of 8.2, 0.59 % organic matter, 229 mg kg^{-1} available K, 12.1 mg kg^{-1} available P, DTPA extractable Fe, Mn, Zn and Cu concentration were 1.65, 8.14, 0.32 and 0.62 mg kg^{-1} and available B with hot water extractable was 0.78 mg kg^{-1}. This experiment consisted of 20 treatments and 3 replications in the form of completely randomized block design and factorial that combinations of five levels Zn (0, 8, 16 and 24 kg ha^{-1} Zn and Zn foliar spray) and four levels of B (0, 3, and 6 kg ha^{-1} and B, and B foliar spray). Nitrogen: P: K used at 180, 70 and 75 kg ha^{-1} according to the recommendation, from sources of urea (46% N), triple super phosphate (46% P_2O_5) and potassium sulfate (50% K_2O), respectively, were added to all treatments (plots). Moreover, 50% of the urea was used when planting and the remainder two times: At vegetative growth (35 days after planting) and when the corn ears were formed. Potassium and P used before planting. Zinc and B, from zinc sulfate and boric acid sources, respectively, were used by two methods, adding to the soil and spraying. Addition to the soil was made at the time of plantation and the sprayings were made at 0.5% Zn sulfate and 0.3% B two times: one at vegetative growth stage and the other after corn ears formation. The Zn and B were both applied to the leaves with uniform coverage at a volume solution of 2500 L ha^{-1} using a knapsack sprayer. Each experimental plot was 8 m length and 3 m width, had 5 beds and 4 rows, equally spaced, and seeds 20 cm apart on the rows. At the end of the growth stage the available Cu and B in the soil after corn harvest were measured.

The soil samples were air-dried and ground to pass through a 2-mm sieve before analysis. Selected soil chemical and physical characteristics for the site are presented in Table 1. Analysis of the soil was carried out using common lab procedures (Soil and Plant Analysis Council, 2004). Soil particle size distribution was determined by the hydrometer method (Gee and Bauder, 1986), organic matter (OM) content by the Walkley-Black method and pH was determined at a 1:2.5 soil/water ratio. Soil available K was determined by 1 M NH_4OAc extraction and K assessment in the extract by flame photometer (Thomas, 1982). Soil P available was measured by Olsen method. Available Fe, Zn, Mn and Cu in the soil were first extracted by DTPA and then were read by atomic absorption. The soil's available B was extracted by hot water and then was measured by spectrophotometer by curcamin method, considering the intensity of the color produced. Each variable was subjected to ANOVA using the Statistical Analysis System (SAS version 8.2, SAS Institute, 2001) for each soil. Treatment (fraction) means were separated by Duncun's multiple range test ($P < 0.05$ level). Multiple regression analyses (stepwise procedure) (SAS Institute, 2001) was conducted to evaluate the relationships between residual available Cu and B in the soil and other factors.

Result and Discussion

Soil analysis result before culture

Physicochemical characteristics of soil taken before the experiment was initiated in the spring of 2009 are presented in Table 1. While table 1 indicates the soil available P was low but available K was higher than the critical level suggested in scientific sources (Karimian and Yasrebi, 1995). Karimian and Ghanbari (1990) have reported the critical P level by the Olsen method in calcareous soils as 18 mg kg^{-1}. The soil Zn, B, Fe and Cu contents were lower but available Mn was higher than the critical level. High soil pH and CaCO3 content induce B deficiency in the surveyed area. Similar results were found by Borax (1996) and Rashid et al. (1997). In soil, the B concentration of <0.65 mg kg^{-1} and >3.5 mg kg^{-1} are deficient and toxic levels for cotton crop, respectively (Annonymous, 1985). For many crops, a DTPA-extractable Zn level of 0.5-0.8 mg kg^{-1} has been regarded as a soil critical level below which crop production would be limited by Zn deficiency (Martins and Lindsay, 1990). Sims and Johnson (1991) have reported the critical levels of Fe, Zn, Mn and Cu by the DTPA extraction method and B by the hot water in the soil method to be 2.5-5, 0.2-2, 1-5, 0.1-2.5 and 0.1-2 mg kg^{-1}, respectively.

Table 1. Soil mechanical and chemical analysis

Properties	Values
Depth of soil(cm)	0 -30
Soil texture	Loam
pH	8.2
EC (ds m^{-1})	2.41
Organic matter (%)	0.59
P	12.1
K	229
Fe	1.65
Mn	8.14
Zn	0.32
Cu	0.62
B	0.78

Residual available copper in the soil

The soil Cu content before planting was 0.62 mg kg^{-1} and increased after harvesting in all treatments (Table 2). Considering that Cu fertilizer was not applied to the soil, the increase in soil Cu content after harvesting was due to availability of a part of the total Cu in the form of available Cu. Root secretions and reactions was carried out in the soil by activities such as irrigation and climate changes during the growing season leads into availability of a part of the total Cu in the form of available Cu. Also, by adding zinc sulfate fertilizer can increase available Cu content in the soil by replacement of Zn instead of Cu. Therefore, in addition to the presence of total Cu in the soil can meet the plant needs, also, some amount of the total soil Cu content be available to the plant after culture and operations on the soil. Of course, with the elapse of time the total available Cu content in the soil decrease to the unavailable form.

The use of different Zn levels and Zn-B interaction on the residual available Cu in the soil was insignificant at 5% level. No treatments showed significant difference from the control. Yang et al., (2000) reported that the decline in residual values of B from a single fertilizer addition was closely related to the soil and leaf B concentration. There was a relation between the leaf Cu content and the residual Cu content, so that Zn and B application had no significant effect on the leaf Cu content; that is the amount of Cu removal from the soil by plant did not change by Zn and B application and consequently residual Cu in the soil did not change as affected by Zn and B application. Considering that Zn and B application at all levels had no significant effect on the residual Cu content in the soil relative to the Zn and B levels and also, almost all treatments showed no significant difference from the control, therefore increasing the residual Cu content in the soil depend on the many factors such as soil tillage, irrigation, root secretions and NPK fertilizers.

Several soil properties such as pH, redox potential (Eh), cation exchange capacity, organic matter, texture, oxide content, and clay mineralogy influence the relative distribution of Cu in different chemical forms (McLaren et al., 1983; Sims, 1986). Soil properties, metal characteristics, and environmental factors influence Cu and Zn concentrations and loads in surface soil (Zhang et al., 2003; He et al., 2004).

Table 2. The effect of Zn and B on residual available Cu in the soil after corn harvest (mg kg^{-1})*

B	Zn (kg ha^{-1})					
(kg ha^{-1})	0	8	16	24	Foliar Spray	Mean
0	0.88 ab	0.63 b	0.69 b	0.85 ab	0.93 ab	0.80 ab
3	0.69 b	0.65 b	0.66 b	0.88 ab	0.78 ab	0.73 b
6	0.71 b	0.85 ab	0.96 ab	0.61 b	0.72 b	0.77 ab
Foliar Spray	0.97 ab	0.73 b	0.87 ab	0.80 ab	1.11 a	0.90 a
Mean	0.82 a	0.72 a	0.80 a	0.79 a	0.88 a	

*Means with same letters lack a significant difference ($p < 0.05$) by Duncan's test

Residual available boron in the soil

The B amount before harvesting was 0.78 mg kg^{-1} and decreased after harvesting in all treatments except the three treatments of 3 kg ha^{-1} B (with a residual B content of 1.3 mg kg^{-1}), 6 kg ha^{-1} B (with a residual B content of 0.83 mg kg^{-1}), and joint use of 6 kg ha^{-1} B and 8 kg ha^{-1} Zn (with a residual B content of 0.83 mg kg^{-1}) relative to the its initial level (Table 3). In fact, at no Zn level, the use of B increased residual B in the soil as compared with the its level before culture; but at presence of Zn, B application had no significant effect on the residual B in the soil relative to the its initial level. Reduction of residual B in the soil relative to the initial B has various causes: or B removal by plant has been more than the amount of B in the soil (initial soil B + B fertilizer), or large amount of boric acid was added to the soil, to become unavailable, or small amount of total soil B has been available. Due to high soil pH (calcareous soil) the amount of boron added to the soil as B fertilizer comes unavailable.

Gupta (1993) reported that after the crop was harvested, lower quantities of hot-water-soluble boron were found in the soil. When boron is released from soil minerals, mineralized from organic matter, or added to soils by means of irrigation or fertilization, part of the boron remains in solution, and part is adsorbed (fixed) by soil particles. An equilibrium exists between the solution and adsorbed boron (Gupta, 2007). Usually more boron is adsorbed by soils than is present in solution at any one time, and fixation seems to increase with time (Jame et al., 1982). Soil factors affecting availability of B to plants are: pH, texture, moisture, temperature, organic matter and clay mineralogy (Goldberg, 1997). Soil reaction or soil pH is an important factor affecting availability of boron in soils. The availability of boron to plants decreases sharply at higher pH levels, but the relationship between soil pH and plant boron at soil pH values below 6.5 does not show a definite trend (Barker and Pilbeam, 2007).
Boron retention in soil depends upon many factors such as the boron concentration of the soil, soil pH, texture, organic matter, cation exchange capacity, exchangeable ion composition, and the type of clay and mineral coatings on clays (Gupta, 2007).

Table 2. The effect of Zn and B on residual available B in the soil after corn harvest (mg kg^{-1})*

B (kg ha^{-1})	Zn (kg ha^{-1})					
	0	8	16	24	Foliar Spray	Mean
0	0.42 b	0.33 b	0.45 b	0.30 b	0.33 b	0.37 b
3	1.30 a	0.63 b	0.78 b	0.35 b	0.39 b	0.69 a
6	0.83 b	0.83 b	0.71 b	0.43 b	0.45 b	0.65 a
Foliar Spray	0.36 b	0.40 b	0.38 b	0.37 b	0.37 b	0.38 b
Mean	0.73 a	0.55 ab	0.58 ab	0.36 b	0.38 b	

*Means with same letters lack a significant difference ($p< 0.05$) by Duncan's test

The effect of different Zn levels on the residual B in the soil was significant at 5% level. The highest mean residual B in the soil, 0.73 mg kg^{-1}, was seen at no Zn level. The use of 16 and 24 kg ha^{-1} Zn, showed no significant difference from the no Zn level, but application of 24 kg ha^{-1} Zn and Zn solution spray decreased residual B in the soil from 0.73 at no Zn level to 0.36 and 0.38 mg kg^{-1}, respectively (50.7 and 48 percent decrease as compared with the no Zn level). There was no significant difference between the Zn spraying and applying Zn to the soil. No Zn content in the soil helped increasing residual B in the soil relative to levels of Zn applied to the soil.

The main effect of B on the residual B in the soil was significant at 5% level. At no B and spraying B levels where B fertilizer was not applied to the soil, the soil B content after harvesting (0.37 and 0.38 mg kg^{-1}, respectively) was less than that of other levels. The use of 3 and 6 kg ha^{-1} B increased residual B in the soil from 0.37 at no B level to 0.69 and 0.65 mg kg^{-1}, respectively (86.5 and 75.7 percent increase relative to the no B level), but no significant difference was seen between these two B levels. The minimum residual B in the soil at 0.37 mg kg^{-1}, was seen at no B level.

At no Zn level, only application of 3 kg ha^{-1} B increased residual B in the soil from 0.42 to 1.3 mg kg^{-1} (209% increased relative to the no B use at this Zn level), but other B levels had no significant effect. At other Zn levels, B use had no significant effect on the residual B in the soil as compared with no B use at these Zn levels.

At 3 kg ha^{-1} B level, Zn application at all levels (to the soil and spraying), significantly decreased residual B in the soil, but at other B levels, Zn application had no significant effect on the soil B content relative to the no Zn use at these B levels. At 3 kg ha^{-1} level, the use of 8, 16 and 24 kg ha^{-1} Zn decreased residual B in the soil from 1.3 to 0.63, 0.78 and 0.35 mg kg^{-1}, respectively (51.5, 40 and 73 percent decrease relative to the no Zn use at this B level). Also Zn spraying at 3 kg ha^{-1} B level, decreased residual B in the soil from 1.3 to 0.39 mg kg^{-1} (70% decrease relative to no Zn use), but showed no significant difference when Zn was applied to the soil.

No treatments, except the treatment with the highest residual B in the soil (application of 3 kg ha^{-1} B) had no significant difference on the residual B in the soil from the control. Application of 3 kg ha^{-1} B, with a residual B in the soil of 1.3 mg kg^{-1}, showed 209 percent increase relative to the control, with a residual B in the soil of 0.42 mg kg^{-1}. In the control which did not use Zn and B, residual B in the soil decreased 48 percent relative to the initial soil B amount (0.81 mg kg^{-1} B).

The correlation between the residual available Cu and B in the soil with other variables

The correlation coefficients (r) between different variables by the Pearson method and the relevant equations were obtained by the step by step method using the SPSS software. One can use each of the following equations depending on what are the variables measured and r and r^2, but the last equation derived, is the most complete equation containing dependent and independent variables and we must measure more variables to derive that equation. The symbols * and ** in equations and correlation coefficients (r or r^2), are significance at 5% $(\alpha = 0.05)$ and 1% $(\alpha = 0.01)$ levels.

The residual available Cu content in the soil

There was a positive correlation between residual Cu in the soil and residual P (r = 0.43) and Fe (r = 0.84^{**}) in the soil, leaf Zn content (r = 0.33), grain Zn content (r = 0.37), the percentage of grain in the ear (r = 0.69^{**}), and grain protein content (R= 0.37), and a negative correlation with leaf Mn content (r = -0.36), grain Cu content (r = - 0.58^{**}) and Cu uptake by the grain (r = - 0.47^{*}). The relevant equations were:

1) CuS = 0.174 + 0.157 FeS r = 0.84^{**}

2) CuS = 0.253 + 0.155 FeS – 0.14 BS $r^2 = 0.78^{**}$

CuS, FeS and BS are residual Cu, Fe and B in the soil (mg kg^{-1}), respectively.

The residual available B content in the soil

The residual B in the soil showed a positive correlation with the residual Mn in the soil (r = 0.37) and leaf B content (r = 0.36) and a negative correlation with leaf N content (r = - 0.54^{*}) and P content (r = -0.35). The equation was:

BS = 3.232 – 1.136 NL r = 0.54^{**}

BS and NL are residual B in the soil (mg kg^{-1}) and N concentration in the leaf (%), respectively.

Conclusion

The residual Cu in the soil increased in all treatments relative to its initial level. The effect of Zn and B on the residual Cu in the soil was insignificant relative to the no Zn and B level. Also the Zn and B interaction on the residual Cu in the soil and the effect of all treatments relative to the control was insignificant. The residual B in the soil in all treatments decreased relative to the soil B content before planting. At no Zn level, B application to the soil increased residual B in the soil as compared with its initial level. Therefore, the presence of Zn in the soil prevented from increase of the residual B in the soil, by B application. The main effect of Zn and B on the residual B in the soil was significant relative to the no Zn and no B levels. The highest and the lowest residual B in the soil were seen at no Zn and B levels, respectively. Application of Zn at a high amount decreased residual B in the soil relative to the no Zn level. Boron application to the soil at all levels, increased residual B in the soil relative to the no Zn level. many factors such as soil tillage, irrigation, root secretions and NPK, Zn and B fertilizers affected on the residual available Cu and B content in the soil relative to the its initial.

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