Contribution of Eelemental Sulphur to Soil Acidification, Fe Release and Uptake by maize (Zea mays L.)

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***Abstract*—A glasshouse experiment was conducted to elucidate the effectiveness of elemental sulphur as a soil acidulates on solubility of soil Fe and it's uptake by maize (Zea mays L.). Four rates of elemental sulphur, 0, 0.5, 1 and 2 g S kg^{-1} soil, incubated for 0, 20 and 40 days before maize plantation. The result showed that with one unit increase in S application rate the soil pH decreased about 1.52 units and the solubility of the Fe was significantly increased. The concentration of Fe in maize leaves and stem were increased with soil acidification from the background of 7.03 to 5.42 due to elemental sulphur application rate of 1 g S kg^{-1} soil. However, further soil acidification decreased Fe concentration in maize. Overall, application of elemental sulphur at a rate of 0.5 g S kg^{-1} soil is recommended to enhance maize performance by 45 percent without the risk of Fe toxicity for maize and the minimum Fe export to groundwater.**

***Index Terms*—Bintang Series soil, iron solubility, soil acidity.**

I. INTRODUCTION

Highlight Availability of micronutrients in soils largely depends on soil type and environmental factors such as acidity (Rengel et al., 1999; Marschner, 2012). Fe deficiencies generally occur in alkaline and calcareous soils because of their high pH that depress solubility of Zn and Fe in soil and decrease its uptake by roots (Zuo and Zhang, 2011). However, using elemental sulphur may result in acidification of the rhizosphere soil; for every pH unit decrease there is an increase in solubility of Zn and ferric Fe by a factor of 10–1000 (Chen and Barak, 1982). Elemental sulphur, as a soil amendment, is of special interest to increase soil nutrient solubility since it possess the slow release acidifying characteristic and readily available (Chien et al., 2011). The acidifying function of S originates from its microbial oxidation to sulphuric acid over time (Vidyalakshmi et al., 2009). Zuchi et al., (2012) reported that providing S above adequate concentrations may result in the improvement of Fe use efficiency in wheat plants. It is also interesting to note that this S nutritional effect seems to be especially advantageous for plants grown under severe Fe limitation. They found that high S supply increased the concentration of Fe in the shoots. In addition, concentrations of Fe and S in the leaves were significantly correlated, irrespective of Fe availability in nutrient solution. The effect of S nutrition on Fe accumulation can be explained by an increased production of phytosiderophores and nicotianamine possibly due to increased methionine level (Zuchi et al., 2012).

There is contrasting reports on the effect of elemental S on soil pH and nutrient availability (Klikocka, 2011; Safaa et al., 2013; Skwierawska et al., 2012). The effectiveness of elemental sulphur application on nutrient solubility was not observed in some soils (De la Fuente et al., 2008; Sameni et al., 2004; Shenker & Chen, 2005; Skwierawska et al., 2012). At the same time, the positive effect of elemental sulphur on soil nutrient solubility is reported by Cui et al. (2004). The increased release of soil nutrient from unavailable to available pools could be due to soil pH changes as reported by Ye et al. (2010). They also showed the increased plant nutrient availability due to soil pH reduction because of S application. Cui et al., (2004) also reported the pH dependence of mobile fraction of soil heavy metal and their increase with soil pH reduction. Increasing the weathering rate as a result of high concentrations of hydrogen ions under acidic conditions is known as an explanation for increased nutrient availability (Lambers et al., 2008). Protons first displace cations from the exchange complex on clay minerals and soil organic matter. The availability of other ions is strongly affected by pH because this affects their oxidation state and solubility (Lambers et al., 2008).

As different soils may show diverse responses to soil acidification as an effective strategy for soil nutrient solubility enhancement (Wang et al., 2006), it is necessary to find the optimum sulphur rate to obtain optimum pH for each specific soil in which nutrient solubility increased and concurrently extreme soil acidification and its consequences such as nutrient toxicity for plants and nutrient leaching to ground water were avoided. Although the effectiveness of elemental sulphur on soil micronutrient release was elucidated by Karimizarchi et al., (2015), the minimal research data are released on impacts of elemental S addition on Fe uptake by plants grow in Bintang Series soils. So, it is essential to quantify the effect of elemental S on the uptake and distribution of Fe in maize plants under acidified Bintang Series soil.

II. MATERIAL NADM METHODS

To elucidate the effect of soil acidification on soil Fe solubility and uptake by maize, the Bintang Series soil was amended with 0, 0.5, 1 and 2 g elemental sulphur kg^{-1}soil and incubated for 0, 20 and 40 days before maize planting in plastic pots. The maize plants were grown for 45 days under glasshouse conditions. Soil samples were retrieved at planting and harvesting stages and subjected to nutrient analysis. In addition, three maize parts including leaves, stem and root were provided separately and analyzed for nutrients.

A. Site Description and Soil Characterization

Soil samples were collected from the A horizon (0-20 cm) of Bintang Series soil located in Perlis, Malaysia (6° 31′ 01.61″ N and 100° 10′ 12.43″ E). The area, Bukit Bintang is affected by limestone parent materials and is under natural vegetation (Karimizarchi et al., 2014a). Soil samples were air dried and ground (< 2 mm) before use. Soil electrical conductivity and pH was measured in a soil water suspension (10 g soil to 25 ml deionized water) 24 hours after shaking for 30 min on a reciprocal shaker.

B. Plant Growth and Management

Sweet maize (Zea mays L.) seeds, Masmadu, were provided by Malaysian Agricultural and Development Research Institute (MARDI, 2008). Seeds were germinated in laboratory conditions and transplanted into 30 cm (diameter) by 50 cm (height) plastic pots after 24 hours. Each pot contained 10 kg soil and received three plants which were thinned to one within one week. Seedlings were grown for 45 days in greenhouse conditions located in University Putra Malaysia (UPM). By weighing each pot, plants were irrigated daily to maintain 90 percent soil field capacity moisture content. All plants were supplied with fertilizers based on MARDI recommendation; 120 kg N /ha in the form of urea, 60 kg P_2O_5 in the form of triple superphosphate and 40 kg K_2O in the form of muriate of potash (Karimizarchi et al., 2014b).

C. Soil and Plant nutrient Extraction and Determination

As buffered extractants may hinder the effect of S on soil nutrient solubility, the mobile fraction of soil nutrients extracted by $CaCl_2$ (Jones, 2001; Ye et al., 2011). It was centrifuged for 15 minutes at 3000 rpm and filtered. Plant leave, shoot and root tissues were separately washed in deionized water then dried at 65°C and weighed. After grounding, weighed plant tissues were ashed in a muffle furnace at 480°C for about 10 h and dissolved in diluted acid mixture (Jones, 2001). Nutrient concentrations were determined by ICP-OES (Perkin Elmer, Optima 8300).

D. Statistical Analysis

To model the relationship between plant and soil properties the data were subjected to different regression models at probability level of 0.05 with the help of Sigmaplot software. Using SAS 9.1, Anova analysis and Duncan's test at $\alpha = 0.05$ was employed to determine the significance differences among mean treatments.

III. RESULTS AND DISCUSSIONS

Soil pH was greatly affected by sulphur application rates and timing (Table 1). For instance, incubation of soil for 40 days with sulphur application rates of 0.5, 1 and 2 g kg^{-1} soil before planting, decreased the pH from the background of 7.51 to 6.66, 5.45 and 4.8, respectively. In addition, soil pH was significantly affected by growth stages (Table 1).

TABLE I: SOIL PH CHANGES IN RESPONSE TO ELEMENTAL SULPHUR TIMING (0, 20 AND 40 DAYS APPLICATION BEFORE PLANTING) AND APPLICATION RATES (G S KG^{-1} SOIL) AT PLANTING AND HARVEST.

Sulphur rate	Soil pH							
	At planting				At harvest			
	0	20	40	Mean	0	20	40	Mean
0	7.51Aa	7.44Aab	7.42Ab	7.45Aa	6.99Aa	6.92Aa	6.88Aa	6.93Ab
0.5	7.26Ba	6.75Bb	6.66Bb	6.89Ba	6.30Ba	6.23Ba	6.34Ba	6.29Bb
1	7.22Ca	6.27Cb	5.45Cc	6.31Ca	5.35Ca	5.27Ca	5.17Ca	5.26Cb
2	7.34Ca	5.44Db	4.80Db	5.86Da	3.90Db	3.86Db	4.06Da	3.94Db
Mean	7.33Aa	6.48Ab	6.08Ac		5.63Ba	5.57Ba	5.61Ba	

Averaged across timing, soil pH for sulphur application rates of 0, 0.5, 1 and 2 g S kg^{-1} soil decreased from 7.45, 6.89, 6.31 and 5.86 at planting to 6.93, 6.29, 5.26 and 3.94 at harvest, respectively. The dependence of soil pH to incubation time and growth stage shows that oxidation of elemental sulphur is time consuming and that incubation time of 20 days is not enough for complete oxidation of applied S in this study. As it can be seen from the Table 1, there is no significant difference in soil pH between incubation times for all sulphur application rates at harvest. This indicates that elemental sulphur had been totally oxidized to sulphate at harvest under conditions of this experiment.

In order to drive a method for predicting the likely outcome of S additions in Bintang Series soil, the relationship between sulphur rate and soil pH was modelled (Figure 1). Regarding the soil pH at harvest, the relationship between soil pH and sulphur application rate was linear, pH = 6.94 – 1.52 S and $R^2 = 0.98^{**}$. In the other words with each unit increase in S rate, soil pH decreases by around 1.52 units. Averaged across timing, soil pH was 7.03, 6.29, 5.26 and 3.94 for sulphur application rates of 0, 0.5, 1 and 2 g S kg^{-1} soil, respectively. The relationship between S rate and soil pH change is of special interest and needs to be studied for each specific soil. In a laboratory study, Owen et al. (1999) modeled the relationship between elemental sulphur application rate and soil pH. They found that application of 4 tons of S per hectare linearly decreased soil pH from 7 to 4.8. While they reported a slight decrease in soil pH by application of 8 tons of S, compared to S rate of 4 t ha^{-1}, it reached to the minimum of 4.2 at S rate of 12 t ha^{-1}. They found that the relationship between S rate and soil pH was fitted best by exponential model.

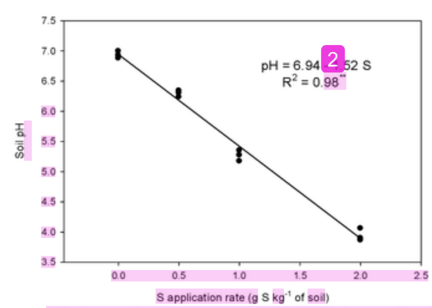

Fig. 1. Soil pH changes in response to elemental sulphur application rate.

As it was outlined above, sulphur addition decreased soil pH and it may affect the release of soil Fe. Therefore, the correlation between soil nutrient availability and soil pH was studied. The results (data not shown) showed the strong and significant correlation between soil pH and soil iron, -0.60**, that indicates the significance of soil pH in soil nutrient release. As the correlation is negative it signifies that with decreasing soil pH the soil Fe release was increased. This is in line with the general opinion of positive effect of soil acidification on soil nutrient solubility (Bolan et al., 2003; Lindsay, 1979; Pendias, 2001; Wang et al., 2006).

A. Soil acidity and Fe solubility

To better understand the pattern of Fe release due to the elemental sulphur management, the bioleaching of soil nutrient as a function of sulphur application rate and timing in Bintang Series soil was elucidated (Table 2). Additionally, as the acidity produced on oxidation of elemental sulphur in soil was known to increase the solubility of micronutrients (Khan et al., 2011), the relationship between soil pH and Fe release for Bintang Series soil was quantified (Figure 2). The results showed that there is no significant change in extractable Fe due to incubation days at planting at each sulphur rate (Table 2). Application of elemental S at 1 and 2 g kg^{-1} significantly increased extractable Fe only at incubation days of 20 and 40. For instance the concentration of Fe at 40 days of incubation significantly increased from 0.11 mg kg^{-1} in unamended soil to 0.21 and 0.24 mg kg^{-1} in soils treated with 1 and 2 g S kg^{-1} soil, respectively. The extractability of Fe also was significantly affected by growth stage. For instance, averaged across timing, the concentration of Fe increased around 4 times from planting to harvest for highest sulphur application rate. The role of soil pH on the solubility of soil et al., 2006). The role of elemental S as an easy to apply release possibility for soil pH reduction and to increase soil Fe nutrients was previously documented (Pendias, 2001; Wang was reported by Shenker and Chen (2005). In line with these, our data showed that with application of elemental sulphur soil pH decreased (Table 1). At the same time, with decreasing soil pH from 7 to 5 the concentration of Fe was slightly affected. However further pH reduction increased Fe solubility in Bintang Series soil under conditions of our experiment (Figure 2). This is in line with the Bolan et al. (2003) observation. They reported the low solubility of Fe even under very acid conditions (Bolan et al., 2003).

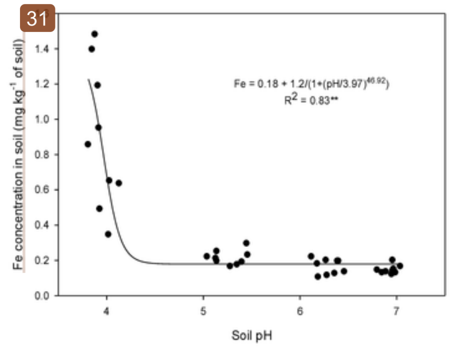

Fig. 2. Soil Fe concentration as function of soil pH.

Our data was fitted with non – linear regression model; $Fe=0.18+ 1.2/(1+(pH/3.97))^{46.92}$, $R^2= 0.83^{**}$. Besides, there was a linear regression model describing the relationship between –Log Fe and soil acidity; $pFe = 0.25\ pH - 0.78$, $R^2= 0.75^{**}$. Although this function is similar to the stability diagrams for Fe as function of pH that developed by Lindsay (1979); Log Fe^{2+} = 15.75 - (pe + pH) - 2pH, however with each unit decrease in soil pH, the LogFe would increase by 0.25 units while that of Lindsay would increase by 2 units. This difference in the rate of Fe change due to soil pH reduction can be attributed to the differences in soil properties as well as the assumptions was considered by Lindsay (1979).

B. Fe as an immobile nutrient in maize

Our data showed that the relationship between elemental sulphur rate and leaf Fe concentration followed a non linear quadratic regression model ($Y= 61.5 +12.58 X-5.85 X^2$ $R^2=0.67^*$). While with increasing elemental sulphur application rate up to 2 g S kg^{-1} soil the Fe concentration in soil was increased (Table 2) its concentration in maize leaves increased up to 1 g S kg^{-1} soil and it decreased at elemental sulphur application rate of 2 g S kg^{-1}(Figure 3). The same trend was found for Fe concentration in maize stem and root (Figure 3). This reflects the fact that maize actively but not passively controls the Fe absorption from the soil solution. Our finding is in line with the Marschner (2012). They stated both passive (carries) and active (coupled transporters) mechanisms for Fe transport across plant membrane. In addition, there is a big difference in Fe concentration in different parts of maize. While the Fe concentration in roots ranged from 500 to 1500 mg kg^{-1}, that of stem and leaves ranged from 30 to 42

TABLE II: SOIL FE CHANGES IN RESPONSE TO ELEMENTAL SULPHUR TIMING (0, 20 AND 40 DAYS APPLICATION BEFORE PLANTING) AND APPLICATION RATES (G S KG^{-1} SOIL) AT PLANTING AND AT HARVEST.

Sulphur rate	Soil Fe (mg kg^{-1} soil)							
	At planting				At harvest			
	0	20	40	Mean	0	20	40	Mean

0	0.21Aa	0.14BCa	0.11Ba	0.15BCa	0.14Ba	0.16Ba	0.13Ba	0.14Ba
0.5	0.14Aa	0.12Ca	0.09Ba	0.12Cb	0.17Ba	0.17Ba	0.14Ba	0.16Ba
1	0.12Aa	0.18Ba	0.21Aa	0.18ABa	0.25Ba	0.18Bb	0.20Bb	0.21Ba
2	0.15Aa	0.25Aa	0.24Aa	0.21Ab	0.94Aab	1.17Aa	0.54Ab	0.88Aa

and 59 to 69 mg kg^{-1}, respectively (Figure 3). This observation refers to the fact that Fe is an immobile element in maize as stated by Barker Pilbeam (2007). With the highest concentration of 69 mg kg^{-1} in leaves at second and third sulphur application rates, comparison of Fe concentration in our maize plants to the adequate Fe level in shoots (50-300 mg kg^{-1}) shows that Fe was not toxic under conditions of our experiment.

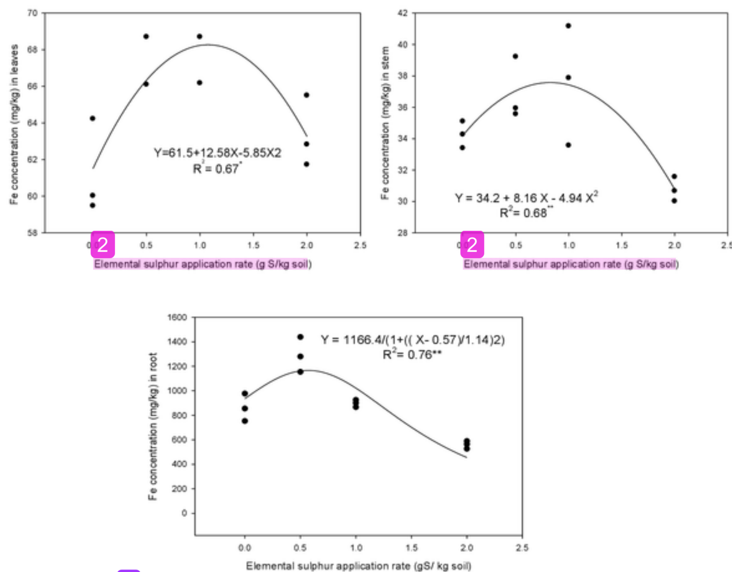

Fig. 3. Effect of elemental sulphur on Fe concentration in different parts of maize.

C. Maize Performance as a Function of Fe Concentration

There is no significant relationship between leaves dry weight and Fe concentration in leaves. In addition, leaves Fe concentration under conditions of our experiment ranged from 60 to 75 mg kg^{-1} (Figure 3). Comparing this values with the recommended adequate range of Fe, 50 to 300 mg kg^{-1} in maize leaves (Barker & Pilbeam, 2007; Campbell, 2000), reveals that this is within the sufficiency range of Fe normally found for maize. However, there is a strong and significant relationship, $Y= 19.3/((1+((X-37.6)/5.4)^2\ ))\ R^2 = 0.695^{**}$, between Fe concentration in stem and stem dry weight. According to this model the maximum performance of maize, 19.3 g pot^{-1}, was obtained at Fe content of 37.6 mg kg^{-1} in stem (Figure 4). As the maximum maize production, in terms of all leaves, stem and root, was not obtained at maximum Fe content that is within the sufficiency range, it can be concluded that the Fe was not the main limiting factor for maize growth under conditions of our experiment. Our conclusion was more supported by the literature review (Bennet et al., 1986; Meriño-Gergichevich et al., 2010).

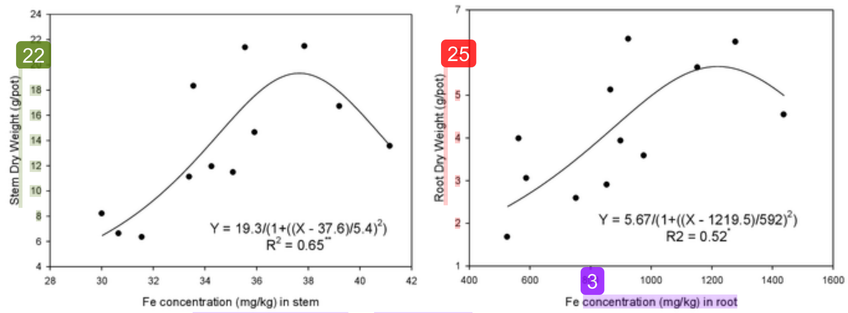

Fig. 4. Relationship between Fe concentration in maize and maize performance.

IV. Conclusion

As soil pH decreased by 1.52 units with each unit addition of elemental sulphur, it can be concluded that elemental sulphur is an appropriate acidulate for Bintang Series soil. In addition, our results demonstrated that the extractable Fe was not significantly affected by soil acidification due to elemental sulphur application unless the soil acidity closed to the pH of hydrous oxide precipitation, around 4, where application of 2 g S kg^{-1} soil increased the $CaCl_2$ extractable Fe by 5.5 times. Moreover, our results showed that this huge increase in soil Fe solubility failed to increase Fe concentration in maize leaves, stem and root. In conclusion, addition of elemental sulphur at a rate of 0.5 g S kg^{-1} soil is recommended for maize performance improvement by 45 percent without the risk of Fe toxicity for maize production and with the minimum risk of Fe export to the groundwater.

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