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Effect of Elemental Sulphur Timing and Application Rates on Soil P Release and Concentration in Maize

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ABSTRACT

High pH soil accounts for more that 30 percent of world's soils and poses problems to plant nutrient availability. As a cheap and readily available source of soil acidulates, elemental sulphur may be a useful material for alleviating some alkaline soil problems. To elucidate the role of elemental sulphur as a soil amendment for plant production in a high pH soil, maize plants were grown under greenhouse conditions for 45 days after 0, 20 and 40 days of soil incubation at different rates of elemental sulphur (0, 0.5, 1 and 2 g S kg^{-1} of soil). Soils were sampled two times (before and after planting) and subjected to soil pH and available P determination. The results showed with each unit increase in S rate, soil pH decreases by 1.52 units. In addition, while sulphur application increased available P before planting, it failed to increase P supply to maize at harvest. Supporting the role of elemental S on soil P availability, with increasing S application rate the P concentration in maize root, stem and leaves was successively decreased. This relationship can be explained by the dilution of P in increasing leaf biomass and the similar concomitant increase of both zinc and manganese nutrient concentrations with increasing sulphur application rate. Overall, soil acidification by elemental sulphur application resulted in P reduction in soil labile pools and intensified P deficiency in maize..

Keywords: Soil acidification, Phosphorous, Mn and Zn

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INTRODUCTION

It is well known that the availability of essential nutrients affects yield and yield components of crops (Ye et al., 2011). The availability of nutrients in soils, as the major source for plant nutrients, depends on soil characteristics especially soil pH (Chien et al., 2011; Lindsay, 1979; Shenker et al., 2005; Wang et al., 2006). Fertilisation and addition of acidifying amendments are common practices in high pH soils to enhance nutrient availability and improve plant performance. Elemental sulphur, as a soil amendment, is of special interest to increase soil nutrient solubility since it possesses slow release acidifying characteristic and is 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). There are contrasting reports on the effects of elemental S on soil pH and nutrient availability (Klikocka, 2011; Safaa et al., 2013; Skwierawska et al., 2012). For instance, the effectiveness of elemental sulphur application on nutrient solubility was not observed in some soils (Sameni et al., 2004; Shenker & Chen, 2005; De la Fuente et al., 2008; Skwierawska et al., 2012). However, the positive effect of elemental sulphur on soil nutrient solubility as a result of soil pH reduction has been well documented (Cui et al., 2004; Ye et al., 2010). As reported by Lambers et al. (2008), high concentrations of hydrogen ions (low pH) cause modest increases in nutrient input by increasing weathering rate. Protons first displace cations from the exchange complex on clay minerals and soil organic matter. In addition, the availability of ions is strongly affected by pH because this affects their oxidation state and solubility (Lambers et al., 2008).

As different soil types 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 were avoided. While the effectiveness of elemental sulphur on Bintang Series soil pH reduction was documented (Karimizarchi et al., 2014), the minimal research data on the impacts of elemental S addition on soil phosphorous release and plant uptake for this soil have been released. Therefore, the present study was carried out to elucidate the effects of elemental sulphur rates and timing, as well as soil acidity on phosphorous solubility in Bintang Series soil and phosphorus uptake by maize. In addition, the phosphorous interactions with Mn and Zn in maize root, stem and leaves are also discussed.

MATERIAL AND METHODS

Site Description and Soil Characterisation

The soil sample for this study was taken from Bukit Bintang, Perlis (located in Malaysia with the geographical coordinates of 6°31′01.61″N, 100° 10′ 12.43″ E). The area, Bukit Bintang, is developed from limestone parent materials and is under natural vegetation (forest). Soil electrical conductivity and pH were measured in a soil water suspension (10 g soil to 25 ml deionised water) 24 hours after shaking for 30 min in a reciprocal shaker. Total carbon, nitrogen and sulphur were determined by CHNS LECO analyser. Meanwhile, soil mechanical analysis was done using the pipette method (Gee et al., 1986) and texture class was determined using the United States Department of Agriculture (USDA) soil textural triangle. Titrimetric method was used for determination of total calcium carbonate (Bashour et al., 2007).

Growth Conditions and Plant Materials

A pot experiment was conducted to elucidate the effects of elemental sulphur application time and rate on maize growth and soil phosphorous release. A completely randomised block design with factorial treatment combination was used with the following factors: (i) Elemental sulphur application at 4 rates including 0, 0.5, 1 and 2 g S per kg of soil; and (ii) elemental sulphur application times including 0, 20 and 40 days before planting of maize. Each pot contained 10 kg soil and received three plants which were thinned to one within one week. The plants were grown for 45 days in the greenhouse. By weighing each pot, the plants were irrigated daily to maintain 90% of soil field capacity moisture content. All the plants were supplied with fertilisers based on the recommendations by Malaysian Agricultural and Development Research Institute; 120 kg N ha^{-1} in the form of urea, 80 kg P_2O_5 in the form of triple superphosphate and 100 kg K_2O in the form of muriate of potash. There were four replications for each of treatments that were randomised in four rows.

Plant Available Soil Nutrient Extraction and Determination

To evaluate the effect of elemental S and soil pH on nutrient solubility, the soluble fraction of soil nutrients was extracted. The mobile fraction of soil nutrients can be extracted by water, neutral or buffered salts (Hlavay et al., 2004; Jones, 2001; Ye et al., 2011). As buffered extractants may hinder the effect of S on soil nutrient solubility, neutral and un-buffered solution, $CaCl_2$ for micronutrients and water for macronutrients were used as five g air dried soil was shaked for 2 hours with 25 ml of 0.01M$CaCl_2$ solution. To obtain a clear solution, it was centrifuged for 15 minutes at 3000 rpm and then filtered. For macronutrients, 10 g air dried soil was shaken for 1 hour with 50 g distilled water. It was centrifuged for 15 minutes at 3000 rpm and filtered. The extracted nutrients were determined by ICP-OES (Perkin Elmer, Optima 8300).

Plant Biomass Nutrient Extraction and Determination

Maize leave, shoot and root tissues were washed separately in deionized water then dried at 65°C and weighed. After grounding, the weighed plant tissues were ashed in a muffle furnace at 480 °C for about 10 h. After cooling, it was dissolved in 10 ml of diluted acid mixture (Jones, 2001). Then, the mixture was filtered into a 50ml volumetric flask through Whatman No. 40 filter paper. Element concentrations including Mn and Zn were determined by ICP-OES (Perkin Elmer, Optima 8300). Phosphorous content of the plant was measured by a Technicon Auto-Analyser.

Statistical Analysis

The relationship between plant and soil properties was subjected to different regression models at a probability level of 0.05 with the help of Sigmaplot software. Using SAS 9.1, Anova analysis and DMRT test at $\alpha = 0.05$ were employed to determine the significant differences between the treatments.

RESULTS AND DISCUSSION

Physico-Chemichal Properties of Bintang Series Soil

With the tentative USDA classification of Ultisol, the physicochemical characteristics of Bintang series soil are presented in Table 1. Being Silt loam in texture, the soil was found to be slightly alkaline in nature with the pH value of 7.5, which is affected by limestone parent materials from nearby hills. Base saturation is high (56 percent), however, the calcium carbonate content of the soil was not detected. Low calcium carbonate content that can be attributed to the high precipitation of the area implies that the soil buffering capacity is low and does not need high amount of acidic soil amendment to reduce soil pH. Supporting our initial assumptions on high pH soils, soil was poor in total carbon, nitrogen and sulphur, with 1.75, 0.12 and 0.004 percent, respectively. This could lead to their shortage for plants.

Table 1
Selected physicochemical properties of Bintang Series soil
(means ± SD, n=3)

Soil property	Unit	Value or Concentration
pH(H_2O)	-	7.51±0.1
$CaCO_3$	%	Tr
Total C	%	1.75±0.05
Total N	%	0.12±0.01
Total S	%	0.004±0.01
C/N	-	16.58± 1.2
C/S	-	437.50± 1.26
CEC	$cmol_+ kg^{-1}$ soil	11.50±0.35
BS	%	56.0 ± 2.0
Texture	-	Silt loam
FC	%	20.00±0.84

Tr: Traces; BS; Base Saturation, FC; Field Capacity, Ex.; Exchangeable

Effects of Elemental S on Soil pH

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

Interestingly, soil pH for treatments not receiving elemental sulphur was significantly different during the growing season. Averaged across timing, the figure was 6.93 at harvest and 7.45 before planting. This can be attributed to low buffering capacity of Bintang series soil, irrigation and fertiliser management and the interactions between soil and plant during the growing season. The soil pH dependence to timing and growth stages for un-treated soil can be attributed to low buffering capacity of Bintang series soil, irrigation and fertiliser management and the interactions between soil and plant during the growing season. This issue was elucidated by Bolan et al. (2003), who reported a decrease in soil pH in soils with low buffering capacity due to generation of H^+ through C, N and S.

In order to drive a method for predicting the likely outcome of S addition in Bintang Series soil, the relationship between sulphur rate and soil pH was modelled (see 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 other words, with each unit increase in S rate, soil pH decreased 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. In line with our results, Owen et al. (1999) reported the linear decrease in soil pH, from 7 to 4.8, by application of elemental sulphur up to 4 tons per ha in a laboratory study. In addition, the relationship between S rate and soil pH for S application range of 0 to 12 tons per hectare was fitted best by exponential model. It should be noted that the relationship between S rate and soil pH change is of special interest and needs to be studied for each specific soil.

Table 2

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 at 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

Means within column followed by the same capital letter and means within rows followed by the same small letter are not significant at the 0.05 level, according to DMRT test at 5% level.

Effects of Elemental S and Soil Acidity on Soil Phosphorous Release

Extractable P was greatly affected by sulphur application rate and maize growth stage (Table 3). Averaged across S timing, application of elemental sulphur increased labile P concentration in soil solution at planting from the background of 0.13 to 0.27 and 0.47 mg kg^{-1} for third and fourth sulphur application rates, respectively. In other words, application of 1 and 2 g S kg^{-1} of soil increased labile P concentration by 145 and 318 percent compared

to the untreated soil at planting. As stated by Ye et al. (2011), the release of P associated with Ca, Al and Fe due to pH reduction could be the primary mechanism by which elemental S application increased P availability at planting. The replacement of PO_4 with SO_4 from exchangeable surfaces is another mechanism responsible for the increased P concentration. However, the increased P availability at planting decreased toward the end of the growing season.

Indicating the transitory effects of elemental sulphur on phosphorous availability, P concentration for untreated soil decreased by 85 percent from planting to harvest and that of treated soils decreased to undetectable amounts at harvest. In line with our finding, the limited long-term effects of sulphur on P availability have been reported by Modaihsh et al. (1989) and Ye et al. (2011). Modaihsh et al. (1989) reported that the incubation of soil with elemental S up to 18 weeks significantly decreased $NaHCO_3$ extractable P. Ye et al. (2011) explained their observation by leaching and runoff, and these two mechanisms can be ignored in the present study as bottom-closed pots were used. This observation can be explained by the conversion of labile P to non-labile forms such as Ca, Al, Mn and Fe bound forms over time and plant uptake (Devau et al., 2009). As concentrations of these elements had increased from planting to harvest (data was not shown), the re-precipitation or adsorption of P by Ca, Mg,

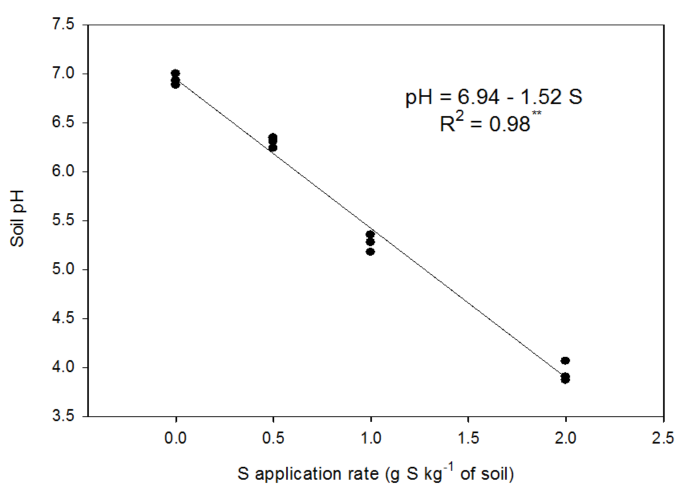

Figure 1. Soil pH changes in response to elemental sulphur application rate.

Mn, Fe and Al could be considered as the possible reason for lower P at harvest in our conditions. The importance of Ca activity on the solubility of P has been stated by Foth and Ellis (1988). They showed that with increasing Ca activity, the solubility of P from rock phosphate had decreased. It is also known that Ca activity may not be closely related to pH. For this reason, the consequences of using rock phosphate even in very acid soils are sometimes unpredictable. Adhami et al. (2007) also reported the association of P with Mn. Using x-ray adsorption near edge structure (XANES) spectroscopy, the existence of Ca phosphates in acid soils and Al phosphates in calcareous soils has been reported by Harrell (2005). This conclusion is more supported by the negative and significant correlation between extractable P and all nutrients, except that of K, Mn and Mg (data were not shown). It indicates the precipitation of P by nutrients such as Ca and Al. The decreased concentration of P with increased S rate at harvest can be attributed to its adsorption by soil minerals as previously described (Lumsdon, 2012). The decrease in available P and exchangeable Ca by soil acidification was reported by Owen et al. (1999). Bolan et al. (2003) also reported the precipitation of P with increases in Fe and Al concentrations due to soil pH reduction. Using geochemical modelling for a better understanding of soil process, Devau et al. (2009) showed that iron-oxides and gibbsite were the predominant P-adsorbing soil constituents at acidic and alkaline conditions, whereas P was mainly adsorbed by clay minerals at intermediate pH values.

In addition, there was no specific relationship between soil pH and P concentration under conditions of our experiment. The complexity of the solubility of P was previously documented by Jones et al. (2005) who had demonstrated that the availability of P is highly dependent upon soil pH and that the maximum availability could be obtained at pH 6.5. In neutral to high pH soils, the available P concentration is largely controlled by the solubility of P minerals that are dominated by calcium phosphates (Ca-P). However, at pH levels below 6, it is controlled by Al and Fe phosphates (Al-P and Fe-P). The poor

Table 3
Soil P 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 P (mg kg $^{-1}$ soil)							
	At planting				At harvest			
	0	20	40	Mean	0	20	40	Mean
0	0.16Ca	0.08Ca	0.15Ba	0.13Ca	Tr	0.04a	0.03Aa	0.02Ab
0.5	0.09Ca	0.11Ca	0.12Ba	0.11Ca	Tr	Tr	0.02A	Tr
1	0.32Ba	0.23Ba	0.27Aa	0.27B	Tr	Tr	Tr	Tr
2	0.54Aa	0.55Aa	0.34Ab	0.48A	Tr	Tr	Tr	Tr

Means within column followed by the same capital letter and means within rows followed by the same small letter are not significant at the 0.05 level, according to DMRT test at 5% level. Tr = traces.

negatively significance correlation between P and Al, Cu, Fe, Zn and Ca concentrations (i.e., less than 0.3), under conditions of our study, demonstrates that the solubility of P was complex and controlled by several factors. This is in line with the findings of De la Fuente et al. (2008), who reported the temporary effect for solid olive mill waste on soil nutrient solubility.

Effects of Elemental Sulphur on Nutrient Concentration in Maize

There is a successive decreasing trend between leaves, stem and root P concentration and S rate (Figure 2). It means that with increasing S application rate, the P deprivation has been intensified. As it can be seen, leaf, stem and root P concentrations varied from the maximum of 0.13, 0.12 and 0.073 percent in untreated soil to the minimum of 0.074, 0.086 and 0.06 percent for the soil treated with 2 g S kg^{-1}. Although there is a decreasing trend between S rate and P concentration in maize, the P concentration in stem and roots had the tendency to increase at highest S rate. This can be because of the profound decrease in dry matter production that can result in the increase of P concentration. Our finding is in contrast with the positive effect of elemental S on P concentrations in maize reported by Kayser (2000).

The decreasing trend in P concentration due to sulphur addition can be related to the interactions of P with other nutrients. For instance, the negative effects of Mn and Zn application on P uptake in common bean had previously been reported in glasshouse experiments (Fageria, 2002). Our results indicate that the increase in Zn concentration in maize tends to result in lower leaf, stem and root P concentration (Figure 3). As clearly shown in the graph, there is a negative, strong and linear relationship between P and Zn concentration in all parts of maize. Therefore, it can be concluded that Zn has an antagonistic effect on phosphorous uptake in maize. This conclusion is more supported by the Zn concentration in maize. The concentration of Zn at the sulphur application rates of 0.5, 1 and 2 g S kg^{-1} soil was 103.63, 121.13 and 166.73 respectively and all are more than the adequate range for maize (20-100 mg kg^{-1}), as recommended by Barker and Pilbeam, (2007). The interactions of P and Zn are diverse and have been previously reported by Fageria (2002). For instance, P-induced Zn deficiency because of the high application rates of P fertiliser to soils low in available Zn has been well documented (Marschner et al., 2012). The researchers proposed that Zinc deficiency increases the permeability of the plasma membrane of root cells to P, as well as to Cl and B, and may even lead to B toxicity. In addition, the negative effect of high Zn concentration on P uptake was also reported by Fageria (2002). He showed that with the application of Zn, P uptake in common bean was decreased (Fageria, 2002).

The high Zn concentration in maize under the conditions of our experiment can be attributed to the release of Zn due to the application of elemental sulphur in Bintang Series soil (Table 4). As clearly presented in Table 4, addition of elemental S at a rate of

0.5, 1 and 2g kg^{-1} increased Zn availability more than 7, 49 and 164 times, respectively.

Indicating the negative interaction between Mn and P, there is a downward trend between P and Mn contents of maize leaves, stem and root (Figure 4). As can be seen, with increasing Mn concentration in stem and leaves (up to 300 and 500 mg kg^{-1} dry weight, respectively), the P concentration decreased. However, with further increase in Mn concentrations (i.e., up to 500 and 800 mg kg^{-1} dry weight in stem and leaves, respectively), P concentration tended to increased. This slight increase in P concentration, in spite of the increased

Table 4
The availability of soil Zn and Mn (mg kg^{-1}) in response to elemental sulphur application rate.

Sulphur rate (g kg^{-1} soil)	Nutrient concentration (mg kg^{-1})	
	Mn	Zn
0	1.61 D	0.030 C
0.5	7.26 C	0.20 C
1	26.67 B	1.47 B
2	73.41 A	4.94 A

†Means within column followed by the same letter are not significant at the 0.05 level, according to Tukey test. Values denoted the means across incubation time.

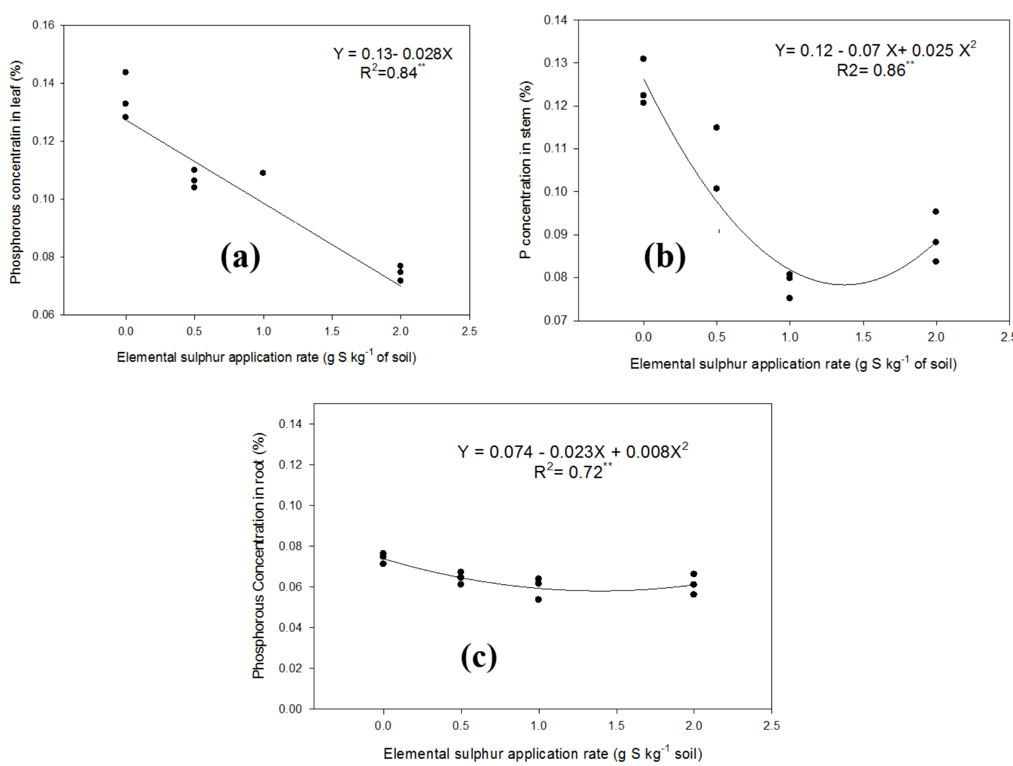

Figure 2. Effect of elemental sulphur on phosphorous concentration in maize leaves (a), stem (b) and root (c).

Mn concentration and its toxicity effects, can be explained by the biomass reduction at highest sulphur application rate synchronised with the highest Mn concentration. The antagonistic effects of Mn and P observed under conditions of our experiment are more supported by the Mn concentration in maize. The concentration of Mn at the sulphur application rates of 0.5, 1 and 2 g S kg^{-1} soil was 81.69, 199.68 and 691.72 respectively, and all are more than the adequate range for maize (50-160 mg kg^{-1}) recommended by Barker and Pilbeam, (2007). The antagonistic effects of Mn and P observed under conditions of our experiment are in line with the previous findings.

For instance, the negative effect of Mn and Al toxicity on P uptake was reported by Bolan et al. (2003). Known as lime-induced P-sparing effect, they reported that soil alkalinisation can decrease Mn and Al toxicity and increase P uptake. It should be noted that the increased Mn concentration in maize leaves, stem and root can be explained by the significant increase in soil Mn concentration due to addition of elemental sulphur (Table 4). As can be seen, with application of elemental sulphur at the rates of 0.5, 1 and 2g kg^{-1}, Mn availability increased more than 4, 16 and 45 times, respectively.

Effect of Elemental Sulphur on Maize growth

Maize leaf, stem and root dry matter production was significantly affected by sulphur application rate (Figure 5). In terms

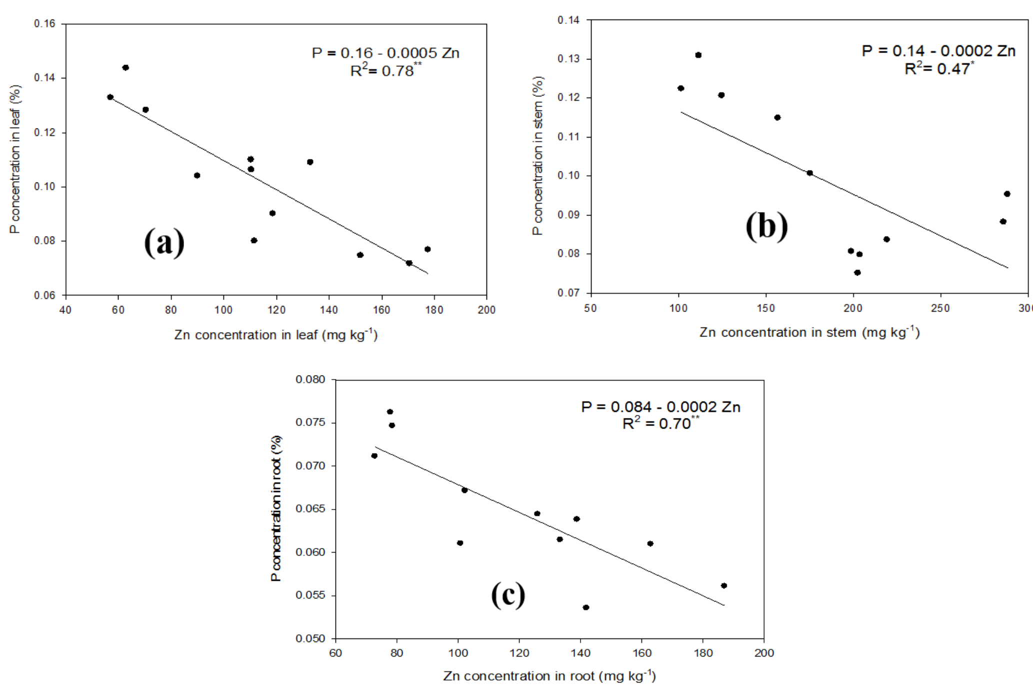

Figure 3. Interaction between P and Zn concentration in maize leaves (a), stem (b) and root (c).

of maize leaves, with the increasing S rate from 0 to 0.5 and 1 g kg^{-1} soil, the leaves biomass production increased by 29.11 and 25.66 percent, respectively. As the leave biomass at S rate of 1 g kg^{-1} soil was equal to 97.32 percent of that at S rate of 0.5 g kg^{-1} soil, it seems that the maximum leave production can be achieved at S application range of 0.5 to 1 g kg^{-1} soil. The similar trend in stem and root production as a function of elemental sulphur application rate was found and illustrated in Figure 5. Being 59 and 44 percent, the increases in stem dry matter production due to application of 0.5 and 1 g S kg^{-1} soil are greater than leaves. Interestingly, the increases in the root production were also found to be greater than stem production, with 81 and 69 percent for S rates of 0.5 and 1 g kg^{-1} soil compared to the un-treated soil.

The negative effect of sulphur application becomes severe at S rate of 2 g kg^{-1} soil, where the maximum sulphur had been applied. This is mainly due to the fact that there is P deficiency in all the treatments. Thus, it should be noted that the value of P is smaller than 0.3 percent, indicating a P deprivation (Barker et al., 2007).

Based on the results of soil analysis (Karimizarchi et al., 2014), the deficiency of P in plants grown in Bintang Series soil was previously predicted. Therefore, the soil was provided with phosphorous fertiliser. However, it seems that more P fertiliser is needed. As stated by Hinsinger et al. (2008),

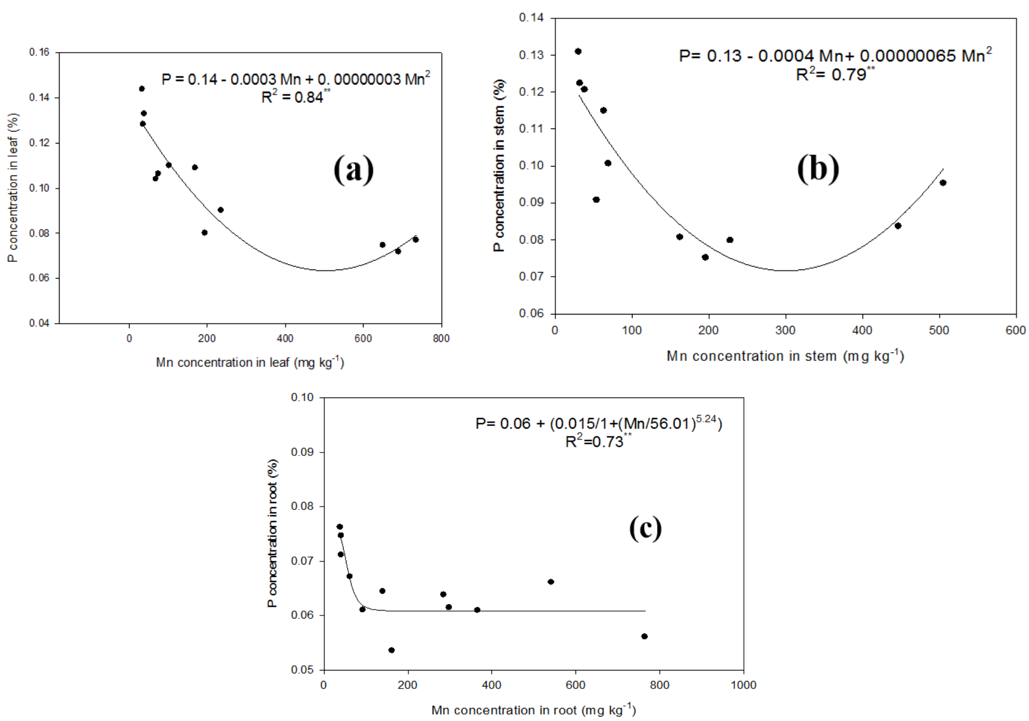

Figure 4. The interaction between Mn and P concentration in maize leaves (a), stem (b) and root (c).

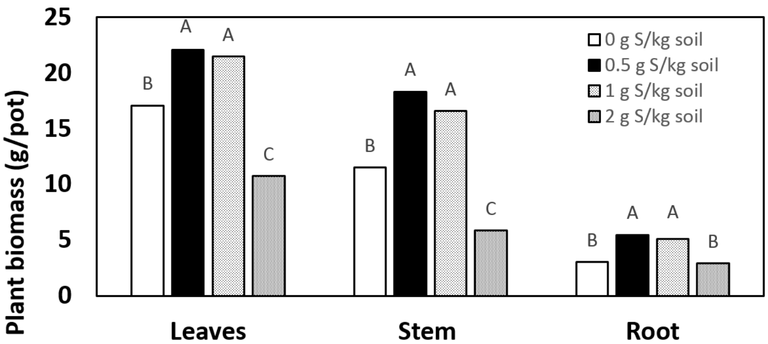

Figure 5. Effect of elemental sulphur application rates (0, 0.5, 1 and 2 g S kg^{-1} soil) on leaf, stem and root dry weight in maize. Means with the same plant part (leaves, stem or root) with a common letter are not significantly different at the 5% level based on DMRT.

the bioavailability of P and K, known as poorly mobile nutrients, depends on the nutrient availability in the soil (including both concentration as well as buffer power). As soil acidification intensified P deficiency in plants and decreased available P in soil, employing foliar application of P can be considered as another option to rapidly alleviate P deficiency. At the same time, enrichment of soil P pools is recommended. This issue can be considered as a future direction.

CONCLUSION

As application of elemental sulphur up to 1 g S kg^{-1} of soil improved maize performance and alleviated S deficiency; thus, it can be used as a soil amendment for crop production. However, as acidification of Bintang Series soil by elemental sulphur decreased available P and reduced P concentration in maize, it can be concluded that the application of elemental sulphur should be accompanied by external sources of P fertilisers for maximising maize production.

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