ORIGINAL PAPER

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Impacts of elemental S applied under various temperature and moisture regimes on pH and available P in acidic, neutral and alkaline soils

Abstract We evaluated the effect of elemental S (S^0) under three moisture (40, 60, 120% water-filled pore space; WFPS) and three temperature regimes (12, 24, 36°C) on changes in pH and available P (0.5 N $NaHCO_3$-extractable P) concentrations in acidic (pH 4.9), neutral (pH 7.1) and alkaline (pH 10.2) soils. Repacked soil cores were incubated for 0, 14, 28 and 42 days. Application of S^0 did not alter the trends of pH in acidic and neutral soils at all moisture regimes but promoted a decrease in the pH of alkaline soil under aerobic conditions (40%, 60% WFPS). Moisture and temperature had profound effects on the available P concentrations in all three soils, accumulation of available P being greatest under flooded conditions (120% WFPS) at 36°C. Application of S^0 in acidic, neutral and alkaline soils resulted in the net accumulation of 16.5, 14.5 and 13 µg P g^{-1} soil after 42 days at 60% WFPS, but had no effect under flooded conditions. The greatest available P accumulations in the respective soils were 19, 19.5 and 20 µg P g^{-1} soil (equivalent to 38, 41, 45 kg P ha^{-1}) with the combined effects of 36°C, 60% WFPS and applied S^0. The results of our study revealed that oxidation of S^0 lowered the pH of alkaline soil ($r=$ −0.88, $P<0.01$), which in turn enhanced available P concentrations. Also, considering the significant relationship between the release of sulphate and accumulation of P, even in acidic soil ($r=0.92$, $P<0.01$) and neutral soil ($r=0.85$, $P<0.01$) where the decrease in pH was smaller, it is possible that the stimulatory effect of sulphate on the availability of P was due to its concurrent desorption from the colloidal surface, release from fixation sites and/or mineralization of organic P. Thus, in the humid tropics and irrigated subtropics where high moisture and temperature regimes are prevalent, the application of S^0 could be beneficial not only in alleviating S deficiency in soils but also for enhancing the availability of P in arable soils, irrespective of their initial pH.

Introduction

The S deficiency in soils in several parts of the world led to the use of fertilizer S to enhance the production and quality of crops (Scherer 2001; Aulakh 2003). Among S-containing fertilizers, elemental S (S^0) is becoming increasingly popular in field crops, including tea gardens that are grown on the steep topographic lands receiving 2,500–3,000 mm rainfall annually. Use of S^0 helps to reduce leaching and run-off losses, leaving prolonged residual effects on the S nutrition of the succeeding crop (Boswell and Friesen 1993). The biochemical oxidation of S^0 produces H_2SO_4, which decreases soil pH and solubilizes $CaCO_3$ in alkaline calcareous soils to make soil conditions more favourable for plant growth, including the availability of plant nutrients (Lindemann et al. 1991). Thus, application of S^0 to alkaline-calcareous soils could assist in correcting iron chlorosis (Saroha and Singh 1980). Soil pH is known to regulate bioactivity and availability of nutrients to plants, because H^+ protons are involved in chemical equilibria. The use of S^0 in alkaline soils reduces soil pH, which may create favourable conditions for the availability of plant nutrients, especially P (Deluca et al. 1989). However, with the decrease in pH of acidic soil, the concentration of water-soluble Fe and Al increases and could cause Fe and Al toxicity. However, information on the use of S^0 in acidic and neutral soils causing changes in soil pH and available P concentrations is lacking.

Of the influencing factors, temperature and moisture are the main factors affecting both the availability of P in soils and changes in soil pH. As the major fraction of soil P is

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present as organic phosphates, temperature and moisture can be important in influencing the amount of extractable P through their effects on microbial activity. Sulphur fertilization is another factor that could influence pH and the availability of P in soils. Sulphate ions can replace and release phosphate ions from the fixation sites, especially where Fe and Al fix substantial amounts of applied P in acidic soils (Pasricha et al. 1987) and calcium binds phosphates in neutral and alkaline soils (Aulakh et al. 2003). The comparative effects of different moisture and temperature regimes (especially the flooded soil conditions and high temperatures prevalent in the tropics and subtropics) and their combined effects with applied S^0 on soil pH and the available P status of soils have seldom been investigated. Such information is needed to understand better the dynamics of P availability in different soils and to predict the effects of applied S^0 on pH-induced changes in plant nutrients for its wider use as an S fertilizer.

In our efforts to develop efficient nutrient management technology for sustainable agricultural management systems for semiarid subtropical regions, we observed that the actual period required for the oxidation and availability of S^0 to plants was determined by prevailing soil pH, moisture and temperature, and that the rates of oxidation of applied S^0 were greatest at 60% water-filled pore space (WFPS) and 36°C, irrespective of soil pH (Jaggi et al. 1999; Aulakh et al. 2002). Thus, the application of S^0 to irrigated crops would be beneficial, but not for flooded rice crop where S^0 does not oxidize because of anaerobic conditions. Also the application of S^0 to summer crops is expected to be more beneficial than to winter crops when temperatures remain quite low. In addition, S oxidation was higher in alkaline than acidic soils. Here, we report the interacting effects of S^0 application in conjunction with different moisture and temperature regimes on the changes in soil pH and available P status over different periods of incubation in acidic, neutral and alkaline soils.

Materials and methods

Soils and preparation of elemental S

Acidic, neutral and alkaline calcareous soils were collected from A horizons of three benchmark sites representing, respectively, the hilly, foothill and plain agro-climatic zones of north-western India. The bulk soil samples were air-dried, crushed, sieved through 2 mm and analysed for important characteristics (Table 1). To ensure that the S^0 applied was practical in terms of its use as a fertilizer, prilled S^0 was ground to pass through a 0.1-cm sieve and retained by 0.05-cm sieve, giving an average diameter of 0.075 cm (Jaggi et al. 1999).

Experimental design and soil analysis

The study was carried out in two phases. In experiment I, treatments consisted of three soils, three moisture regimes (40, 60, 120% WFPS) and two rates of S^0 (0 µg, 500 µg g^{-1} soil). All analyses were done in duplicate and incubated for 14, 28 and 42 days at a constant temperature of 24°C. WFPS, synonymous with relative saturation, incorporates both soil water content and bulk density and is a better index of aeration-dependent biological processes than soil water content (Linn and Doran 1984; Doran et al. 1990). While aerobic soil conditions were simulated by 40% and 60% WFPS, flooded soils (such as those used for rice production) with a thin layer of overlying surface water were simulated using 120% WFPS.

Taking into account that the optimum rate of S^0 oxidation was obtained at 60% WFPS in each of the soils used in experiment I, the individual and combined effects of temperature regimes and applied S^0 were evaluated in experiment II using this optimum moisture regime. Experiment II consisted of three soils, three temperature regimes (12, 24, 36°C) and two rates of S^0 (0 µg, 500 µg g^{-1} soil). As before, all analyses were done

Table 1 Site and important physico-chemical characteristics of the soils from north-western India used in this study

Soil property	Acidic soil	Neutral soil	Alkaline soil	Method
Location, District, State	Taragarh, Kangra, Himachal Pradesh	Basal, Una, Himachal Pradesh	Dagru, Ropar, Punjab	
Sand, silt, and clay (%)	52, 23, and 25	50, 32, and 18	28, 51, and 21	International pipette method (Piper 1968)
Textural class	Sandy clay loam	Sandy loam	Silty loam	
pH	4.9	7.1	10.2	Soil:water suspension (1:2)
CaCO$_3$ (%)	0.7	1.6	3.8	HCl (Puri 1930)
Organic carbon (g kg^{-1})	16.4	8.7	5.9	Wet acid digestion (Walkley and Black 1934)
Total S (µg g^{-1})	423	364	317	HNO$_3$ digestion (Chaudhary and Cornfield 1966)
Organic S (µg g^{-1})	355	170	105	Digestion-turbidimetry (Chesnin and Yien 1951; Bardsely and Lancaster 1965)
Sulphate S (µg g^{-1})	18	90	178	0.15% CaCl$_2$-turbidimetry (Chesnin and Yien 1951; Williams and Steinbergs 1959)

in duplicate and incubated for 14, 28 and 42 das at 60% WFPS.

Repacked soil cores were prepared by placing pre-moist soil (100 g, oven-dry basis) with or without uniformly incorporated S^0 into 200-ml plastic vials (50 mm i.d., sealed from bottom). The soil in each plastic vial was then hand-compacted to give bulk densities of 1.35, 1.42 and 1.51 g cm^{-3} for acidic, neutral and alkaline soils, respectively. These bulk densities are representative of the natural reconsolidation values for the respective soils. Distilled water was added drop-wise, using a fine-jet pipette, to obtain the desired WFPS. Each vial was covered with a perforated polyethylene sheet (held using a rubber band) to facilitate proper aeration during incubation and to restrict evaporation. Thereafter, vials were incubated at the respective temperature regimes in an incubator. Moisture loss through evaporation (determined gravimetrically), if any, was adjusted at 48-h intervals throughout the incubation.

At the end of each incubation period, the soils were removed from the cores and each core was thoroughly mixed and used for different chemical analyses. Soil pH was determined in a soil:water suspension (1.0:2.5, w/v) using an expanded scale pH meter (ECF) and a glass electrode. Olsen-P concentration in sub-samples of all soils was determined after extraction with 0.5 N NaHCO$_3$ (Olsen and Sommers 1982). Sulphate-S was extracted with 0.15% CaCl$_2$ (Williams and Steinbergs 1959) and determined in the aliquots turbidimetrically (Chesnin and Yien 1951).

Statistical analysis and computations

The experiment was laid down in a completely randomised design and the data on various parameters were analysed statistically using ANOVA (Cochran and Cox 1950). Data are presented as means ± standard deviation (SD) of the replicates. The increase in or accumulation of sulphate-S and Olsen-P in the different treatments was calculated by subtracting their respective values at the beginning of the experiment from the value obtained for a given treatment at a given time-point. Correlation coefficients (r values) between different parameters, such as pH, net accumulated sulphate-S and Olsen-P, were then calculated. The values of accumulated Olsen-P were

translated to kg P ha^{-1} using a plough layer (0–15 cm depth) bulk density of 1.35, 1.42 and 1.51 g cm^{-3} for acidic, neutral and alkaline soils, respectively.

Results and discussion

Changes in soil pH

The incubation of the acidic soil without added S^0 showed a small decrease in pH at 40% WFPS, an initial pH increase of 0.2 units that then fell back to the original pH by 42 days at 60% WFPS and a consistent pH rise at 120% WFPS (flooded conditions) in experiment I (Table 2). At the end of 42 days of incubation, a pH value of 6.1 was recorded under flooded conditions, as against its initial value of pH 4.9. In the case of the neutral soil, pH fluctuated around its original value of pH 7.1 throughout the incubation period. The alkaline soil exhibited a continuous, decreasing trend with increasing incubation period. This pH decrease was conspicuously large under flooded conditions. Flooding of soils causes substantial changes in microbial activity. As a result of flooding-induced changes in soil pH, the increase in the pH of acid soils is due mainly to the reduction of Fe^{3+} to Fe^{2+} [Fe$(OH)_3 + 3H^+ + e^- = Fe^{2+} + 3H_2O$], whereas the decrease in the pH of alkaline calcareous soils is the result of the partitioning of CO_2 [pH $= 6.03 - 0.67 \log pCO_2$] in soil solution (Ponnamperuma 1984).

Addition of S^0 did not alter the trends in pH of either the acidic or the neutral soil at any of the moisture regimes (Table 2). For the alkaline soil, application of S^0 prompted a decrease in pH under aerobic conditions (40%, 60% WFPS) but failed to register any effect under flooded conditions. The H_2SO_4 produced from S^0 oxidation caused a decrease in the pH of the alkaline soil under aerobic conditions. However, there was no change in soil pH under flooded conditions largely due to the absence of S^0 oxidation (Aulakh et al. 2002).

In experiment II, increasing the temperature from 12°C to 36°C did not show any particular trend in the pH changes in the acidic soil. After 14 days, the pH registered an increase of 0.3 units at 12°C, which was reduced to 0.1 units at 36°C and eventually returned to its initial value of pH 4.9 after 42 days of incubation (Table 3). In contrast, incubation of the neutral and alkaline soils showed a

Table 2 Influence of moisture regime (WFPS) with and without S^0 on soil pH in acidic, neutral and alkaline soils incubated for 14, 28 and 42 days (experiment I). LSD (0.05): moisture = 0.3, soil = 0.3, elemental S = 0.2

WFPS (%)	Acidic soil (initial pH 4.9)			Neutral soil (initial pH 7.1)			Alkaline soil (initial pH 10.2)		
	14 days	28 days	42 days	14 days	28 days	42 days	14 days	28 days	42 days
Without S^0									
40	4.9	4.8	4.8	7.1	7.0	7.1	10.2	10.1	10.1
60	5.1	5.0	4.9	7.0	6.8	6.8	10.1	10.0	10.0
120	5.5	5.9	6.1	7.2	6.8	7.0	9.8	9.3	8.8
With 500 µg S^0 g^{-1} soil									
40	4.8	4.7	4.8	7.0	6.9	6.8	10.1	10.0	9.9
60	4.9	4.8	4.8	6.9	6.7	6.7	9.9	9.8	9.7
120	5.5	5.9	6.1	7.2	6.8	7.0	9.8	9.3	8.8

decrease in pH with increasing temperature, with the fall in pH reaching a maximum at 36°C. The drop in pH of these soils could be due to the accelerated decomposition of soil organic C with increasing temperature and the resultant production of H_2CO_3 and CO_2 in soil solution (Ponnamperuma 1984).

Application of S^0 reduced the soil pH to differing degrees in different soils (Table 3); and changes in pH were smallest in the acidic soil. This confirms the results obtained in experiment I (Table 2). The limited acidifying effect of S^0 in the acidic soil may be attributed to the relatively lower rates of S^0 oxidation (Aulakh et al. 2002) and to the high buffering capacity of the acidic soils (Boswell and Friesen 1993) that prevented a decrease in soil pH. The fall in pH at 36°C after 42 days of incubation was significant in neutral (0.6 units) and alkaline soil (0.7 units). Jaggi et al. (1999) observed that the higher the temperature the higher the rate of oxidation of S^0 under aerobic soil conditions. The changes in soil pH seen in experiment II followed the patterns of S^0 oxidation and were also lowest in acidic soil, followed by neutral and were highest in alkaline soil (Jaggi et al. 1999). A linear relationship, together with a highly significant negative correlation ($r=-88$, $P<0.01$), was obtained between soil pH and the increase in sulphate as a result of S^0 oxidation in alkaline soil (Fig. 1). This indicates that the fall in soil pH is proportional to the release of H^+ with S^0 oxidation.

Moisture × elemental S effects on Olsen-P concentrations

Incubation of acidic, neutral and alkaline soils without applied S^0 for 14, 28 and 42 days resulted in a consistent increase in Olsen-P concentrations (an index of P availability) under different moisture regimes (Fig. 2). An increase in P availability was observed with increasing moisture with the highest accumulation of Olsen-P in flooded soil. The net increase in P over the 42-day period compared with the initial values ranged over 5.5–11.5 $\mu g\ P\ g^{-1}$ in acidic soil, 7.5–10.5 $\mu g\ P\ g^{-1}$ in neutral soil and 2.5–10.5 $\mu g\ P\ g^{-1}$ in alkaline soil. The amount of soil water influences microbial activity including mineralization of soil organic P. The optimum for activity is 60% WFPS (Linn and Doran 1984; Doran et al. 1990). No doubt, microbial activity declines due to restricted oxygen
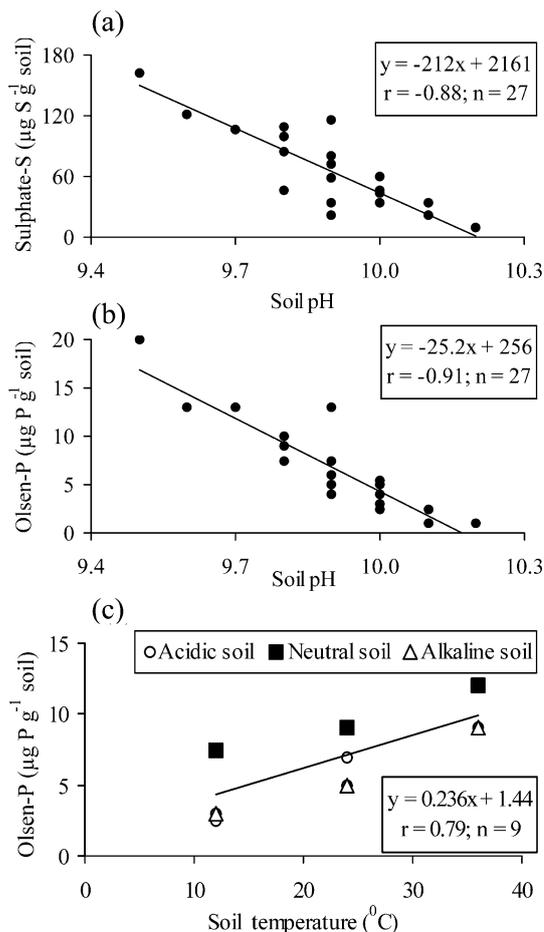
Fig. 1 Relationships between different parameters obtained in soils incubated under aerobic conditions (40%, 60% WFPS) in experiment I and under aerobic conditions (60% WFPS) at different temperature regimes (12–36°C) in experiment II. a Soil pH and accumulation of sulphate in alkaline soil, b soil pH and accumulation of Olsen-P in alkaline soil, c soil temperature and accumulation of Olsen-P in acidic, neutral and alkaline soil

availability at >90% WFPS, which should lead to lower rates of mineralization of organic P. However, upon flooding the soil, phosphate solubility moves towards more soluble phosphate products resulting in increased P availability with an increase in Olsen-P concentrations, apparently due to the dissolution of Ca-P in calcareous and alkaline soils and Al-P and Fe-P in acidic soils (Vig et al. 1999). Thus, the highest concentrations of Olsen-P

Table 3 Influence of temperature regimes with and without S^0 on soil pH in acidic, neutral and alkaline soils incubated for 14, 28 and 42 days (experiment II). LSD (0.05): temperature = 0.3, soil = 0.3, and elemental S = 0.2

Temperature(°C)	Acidic soil (initial pH 4.9)			Neutral soil (initial pH 7.1)			Alkaline soil (initial pH 10.2)		
	14 days	28 days	42 days	14 days	28 days	42 days	14 days	28 days	42 days
Without S^0									
12	5.2	5.0	4.9	7.0	7.0	6.9	10.1	10.0	10.0
24	5.1	5.0	4.9	7.0	6.9	6.8	10.1	9.9	10.0
36	5.0	4.8	4.9	6.9	6.8	6.8	10.0	9.9	9.8
With 500 $\mu g\ S^0\ g^{-1}$ soil									
12	5.0	4.9	4.7	7.0	6.9	6.8	10.0	9.9	9.8
24	4.9	4.8	4.8	6.9	6.7	6.7	9.9	9.8	9.7
36	4.9	4.8	4.7	6.8	6.7	6.5	9.8	9.6	9.5

obtained in each of the test soils at 120% WFPS was the result of the combined interconversion of organic P to inorganic P and the solubilization of insoluble P forms.

Application of S^0 did not alter the trends seen in Olsen-P concentrations in these soils and markedly increased Olsen-P concentrations under aerobic conditions (40%, 60% WFPS). The combined effects of applied S^0 and moisture regimes on the accumulation of Olsen-P over the 42-day period was 8.5 μg and 16.5 μg P g^{-1} in acidic soil, 12.0 μg and 14.5 μg P g^{-1} in neutral soil and 7.5 μg and 13.0 μg P g^{-1} in alkaline soil at 40% and 60% WFPS, respectively. Under flooded conditions, there was a negligible effect of S^0 on Olsen-P in soils and the curves with S^0 overlaid those without S^0 in all three soils (Fig. 2). As was the case with soil pH, the lack of change in Olsen-P concentration with the application of S^0 under flooded conditions was most likely due to the absence of S^0 oxidation (Aulakh et al. 2002).

Temperature × elemental S effects on Olsen-P concentrations

As with the effects of increasing moisture, increasing temperatures were found to result in a significant increase in available P in all soils (Fig. 3). The highest increase in Olsen-P content was obtained at 36°C. After 42 days, the net increases in Olsen-P concentrations due to temperature regimes in the acidic, neutral and alkaline soils were 2.5–8.5 μg, 7.5–12.0 μg and 1.0–9.0 μg P g^{-1} soil, respectively.

As shown in experiment I, the application of S^0 did not change the trends in Olsen-P concentrations in any of the soils. However, there was a marked increase in the Olsen-P concentrations at all temperatures. The use of repacked soil cores at a plough layer bulk density of 1.35, 1.42 and 1.51 g cm^{-3} for acidic, neutral and alkaline soils, respectively, allowed the translation of the concentration values of accumulated available P to kg P ha^{-1}. The combined effect of S^0 and temperature regime on the

accumulation of Olsen-P over 42 days at 60% WFPS was 8.5–19.0 μg P g^{-1} (17–38 kg P ha^{-1}) in acidic soil, 12.5–19.5 μg P g^{-1} (26–41 kg P ha^{-1}) in neutral soil and 7.5–20.0 μg P g^{-1} (17–45 kg P ha^{-1}) in alkaline soil. Increasing temperature stimulated S^0 oxidation (Jaggi et al. 1999), which presumably triggered the release of P. The linear relationship between temperature and Olsen-P concentration in all soils suggests that the availability of P would increase by 0.24 μg g^{-1} with every 1°C increase in temperature (Fig. 1).

Interrelationships between S^0 oxidation, pH and the accumulation of Olsen-P

Deluca et al. (1989) observed a decrease in soil pH as a result of applied S^0 and noted that this could improve the availability of P in alkaline calcareous soils. Jaggi et al. (1999) and Aulakh et al. (2002) identified 36°C and 60% WFPS as the optimum levels for S^0 oxidation in acidic, neutral and alkaline soils from north-western India. In the present study, the relationships between the different parameters under aerobic conditions (40%, 60% WFPS) in experiment I and under aerobic conditions (60% WFPS) at different temperature regimes (12–36°C) in experiment II revealed a highly significant negative correlation between the net accumulation of sulphate-S (as a result of S^0 oxidation) and pH ($r=-0.88$, $P<0.01$) and a highly significant positive correlation between pH and Olsen-P concentration ($r=-0.93$, $P<0.01$) in alkaline soil (Fig. 1). Thus, higher oxidation of S^0 increased the availability of P by lowering pH in the alkaline soil. However, a strong relationship was also seen between the release of sulphate and the accumulation of Olsen-P, even in the acidic ($r=0.92$, $P<0.01$) and neutral soil ($r=0.85$, $P<0.01$; Fig. 4) where the drop in pH was smaller (Tables 1, 2) and showed either no relationship or a negative non-significant relationship with extractable P ($r=0.13$). This suggests that sulphate had a stimulatory effect on the release of Olsen-P. The positive relationship between sulphate-S and Olsen-P

Fig. 2 Effect of different moisture regimes expressed as WFPS with and without S^0 on Olsen-P concentration in acidic, neutral and alkaline soils in experiment I. Note that the curves for 120% WFPS without S^0 are not visible as they are overlaid by the curves of 120% WFPS with 500 μg S^0 g^{-1} soil. *Vertical bars* indicate Standard deviations

Legend: —○— 40% WFPS, without S° · —△— 60% WFPS, without S° · —□— 120% WFPS, without S° · —×— 40% WFPS, 500 μg S° g-1 · —▲— 60% WFPS, 500 μg S° g-1 · —■— 120% WFPS, 500 μg S° g-1

Acidic soil / Neutral soil / Alkaline soil

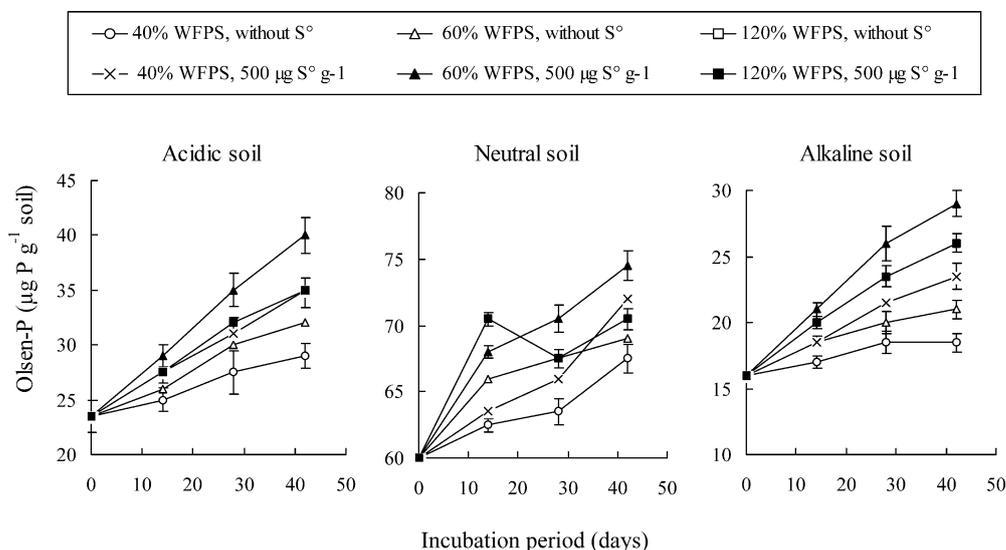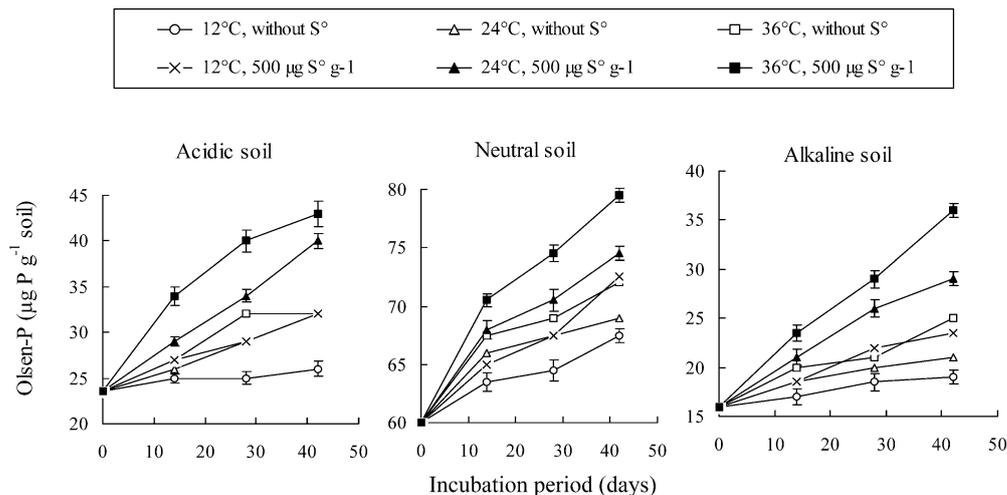Olsen-P (μg P g^{-1} soil)

Incubation period (days)

Fig. 3 Effect of temperature with and without S^0 on available P concentration in acidic, neutral and alkaline soils in experiment II. *Vertical bars* indicate Standard deviations

concentrations in all soils was due both to the decrease in soil pH during S^0 oxidation and to the stimulatory effect of sulphate ions. Increasing the temperature from 12°C to 36°C and moisture from 40% to 60% WFPS optimized the conditions for S^0 oxidation. Undoubtedly, the significant positive relationship between sulphate-S and Olsen-P could be casually linked to decreased pH resulting from the oxidation of S^0. However, it may also indicate a proportional enhancement in the release of P by its desorption from exchange sites by sulphate ions, from mineralization of organic P and/or from fixation sites. Further investigations are, thus, needed to enhance our understanding of the exact mechanisms of P release during the oxidation of S^0.

Conclusions

The results of the present study show that S^0 oxidation is related to moisture and temperature and strongly modifies soil pH and available P concentrations. This could have strong practical implications. The decrease in soil pH was greatest in alkaline soil without S^0 under flooded conditions and in alkaline soil with S^0 at 60% WFPS, where oxidation of the S^0 was rapid, thus improving soil conditions for the availability of nutrients. Smaller acidifying effects were seen with S^0 in acidic soil, which has the advantage that any detrimental effect of applied S^0 on plant growth would be negligible.

The linear relationship between temperature and Olsen-P concentrations in all soils suggests that the availability of P would increase by 0.24 µg P g^{-1} soil with every 1°C increase in temperature. Similarly, the greatest increase in available P (38, 41, 45 kg P ha^{-1} in acidic, neutral, alkaline soils, respectively) obtained with the integrated effects of hydrothermal regimes (60% WFPS, 36°C) and applied S^0 suggests that the agronomic performance of S^0 fertilizers would vary widely with climatic conditions. When climatic conditions are optimal, substantial amounts of P will become available following the application of S^0. Thus, the use of S^0 would be more beneficial in the humid
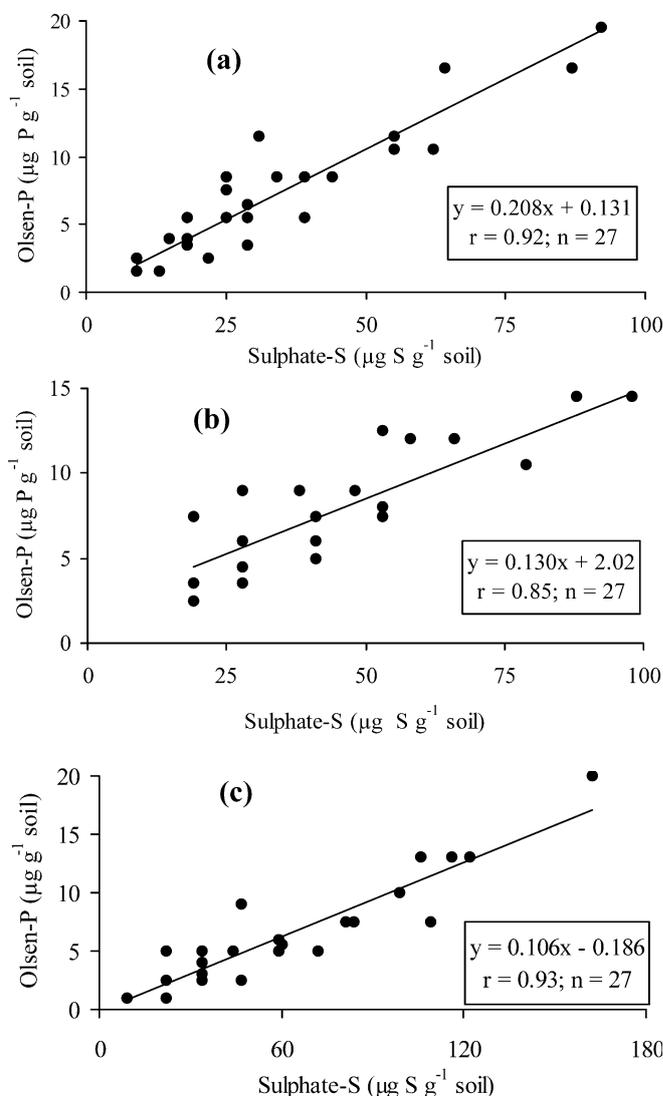

Fig. 4 Relationship between the accumulation of sulphate and Olsen-P in soils incubated under aerobic conditions (40%, 60% WFPS) in experiment I and under aerobic conditions (60% WFPS) at different temperature regimes (12–36°C) in experiment II. **a** Acidic soil, **b** neutral soil, **c** alkaline soil

tropics and irrigated subtropics where both moisture (rainfall or irrigation) and temperature are high. However, as the water content in agricultural fields would vary from dry to saturated and would be expected to be optimal (60% WFPS) after irrigation and rainfall events, the accumulation of available P may be lower under field conditions.

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