CHAPTER 4

SULFUR AND PHOSPHORUS SOURCES FOR PASTURES

4.1 INTRODUCTION

The experiment reported in Chapter 3 showed that the grain yield of flooded and nonflooded rice was significantly influenced by different sources of S. Under these conditions, the oxidation of elemental S-based products was affected by the water regimes. The question arises as to how effective these sources are for pastures under leached and non-leached conditions.

Although different sources of S under flooded and non-flooded rice conditions behaved similarly, further studies are required to evaluate the effectiveness of different S sources under different crops and different water managements for a longer period of time.

Many studies have been conducted on the effectiveness of S and P sources for pastures (Bolland and Bowden, 1984; Boswell, 1987; Friesen *et al.*, 1987a,b; Chisholm and Blair, 1988a,b,c; Bolland *et al.*, 1989). Several studies have been undertaken to investigate the effectiveness of elemental-S-containing products, as well as sulphate-based materials, for pastures under glasshouse as well as under grazing conditions (Shedley, 1982; Blair, 1987a; Boswell, 1987; Bowdler and Pigott, 1990). These results have generally shown that sulphate-containing products, such as gypsum, contribute sulphate to growing crops more quickly than elemental S. However, higher leaching S losses are generally recorded from gypsum in areas with high rainfall and soil with low adsorption capacity (Blair, 1979; Morris, 1987).

Greater quantities of high-analysis fertilisers, such as urea and triple superphosphate, are now produced by the fertiliser industries because of the cost advantage in transportation and application (Suryosunarko, 1989). The increasing use of high-analysis fertilisers, which contain none or only a small amount of S, and a declining use of conventional single superphosphate are the main reasons for increasing sulfur deficiency (Lefroy, 1989).

Phosphate rock has been reported to be an effective source of P for crops in some areas, particularly when soil pH is low (Rajan, 1987). In contrast, many investigators report that this material was not an effective P source due to the slow release of P (Bolland *et al.*, 1989;

Bolland and Gilkes, 1990). A greater acidulation of rock phosphate has been reported when this material was mixed with elemental S (Rajan, 1987) and this might be used to increase its effectiveness. However, the satisfactory performance of the combination of rock phosphate and elemental S will depend on intimate contact between the elemental S and rock phosphate constituents (Palmer and Gilkes, 1983). This experiment was undertaken to study the effects of different S and P sources for pastures under leaching and non-leaching conditions.

4.2 MATERIALS AND METHODS

a. Cores

PVC tubes, 16 cm in internal diameter and 13 cm deep, were driven into an S-deficient, unfertilised pasture on a granitic Aquic Haplustalf soil (Soil Taxonomy, 1975) at Uralla, N.S.W. The chemical analysis of the soil is presented in Table 3.1. The cores with the intact pastures were then removed from the field, capped at the base and for the leached treatments the cores were fitted with special leaching bases. The cores were then transported into the glasshouse and maintained at field capacity by daily weighing and watering with distilled water. The original grasses present were cut to approximately 0.5 cm above the soil surface. The cores were then oversown with perennial ryegrass (*Lolium perenne* L.) and white clover (*Trifolium repens* L.) seeds. Shade cloth was used to cover the pots in order to facilitate seed germination. The pots were kept in a glasshouse with minimum and maximum temperatures of 15 and 25 °C.

b. Basal nutrients and ³⁵S labelling

Basal nutrients (Table 4.1) were diluted in distilled water and applied 14 days before the main treatments were applied. On the same day, carrier free ³⁵S obtained from Amersham Australia Pty. Ltd., was diluted with distilled water to give a solution containing 2.93 MBq ml⁻¹. A syringe was used to apply 5 ml of the radioactive solution to the surface of the soil to enable the reverse-dilution method of Shedley *et al.* (1979), as described in Chapter 3, to be used.

		Application		
Nutrients	Source	kg nutrient ha ⁻¹	mg product pot -1	
N	Ca(NO ₃) ₂	10.0	8.7	
к	KCÌ	40.0	52.0	
Mg	MgCl ₂ .6H ₂ O	24.0	17.0	
Zn	ZnCl ₂	2.0	1.7	
Cu	CuCl ₂ .2H ₂ O	0.2	0.17	
Mn	MnCl ₂ .4H ₂ O	1.0	0.9	
Мо	Na2MoO4.2H2O	0.02	0.017	
В	H ₃ ĒO ₃	0.5	0.17	

Table 4.1 Basal nutrient application rates.

c. Experimental design, treatments and statistical analysis

Two randomized block design experiments with three replicates were conducted in the glasshouse, of the Department of Agronomy and Soil Science, The University of New England, Armidale, N.S.W, Australia.

In the main experiment the treatments (Table 4.2) consisted of the factorial combination of 2 S sources (elemental S and gypsum), 2 P sources, triple superphosphate (TSP) and rock phosphate (RP), 2 methods of S and P application (S mixed with granulated TSP or RP and S coated onto TSP or RP). One unfertilised treatment (C) was included in the experiment in order to calculate the percentage of S in the plant that was derived from the fertiliser. Two watering regimes were imposed where the soil in the non-leached (NL) treatment was maintained at or near field capacity by weighing and a leached (L) treatment where an excess of 25 % of water above field capacity was added every watering. The rock phosphate used in this experiment was from North Carolina, USA, with a P content of 14 %. The rock was finely ground prior to the experiment to a particle size less than 400 µm. Before the RP was coated with elemental S, the RP was granulated using a seed coating device and PVA glue (12 % w/v) was used to make granules. The granule size of the RP was made similar to the TSP granules (2 - 4 mm in diameter). The granulated rock phosphate and TSP were then coated with elemental S of particle size < 100 µm using the same device and procedures as in the previous chapter. Polyvinyl alcohol was used as an adhesive material to bind elemental S with the granulated TSP or granulated rock phosphate. Each fertilised treatment received the same amount of S and P at the rates of 40 mg elemental S pot ⁻¹ or 216 mg gypsum pot ⁻¹ (equivalent to 20 kg S ha⁻¹ based on the surface area of the pot) and 402 mg TSP or 574 mg rock phosphate (equivalent to 40 kg P ha⁻¹).

In a subsidiary experiment the effectiveness of different TSP-S sources (Table 4.3) was investigated under non-leaching conditions. This experiment consisted of 5 fertilised treatments: TSP-S with PVA (PVA), TSP-S with slack wax (SW), TSP-S with sodium ligno-sulfonate (LS), TSP + gypsum (G), a commercial TSP-S which was manufactured by Hifert Fertiliser Ltd., Victoria, Australia, and a control (C). These treatments were replicated three times. Data of PVA, G and C were derived from the non-leached treatment in the main experiment. The same computer program as used in Chapter 3 was used in this experiment to analyse the data. Data from control were excluded from the analysis of variance, but the means of these data are presented in each graph for comparison. Data presented throughout this study are from the S × P × L interaction due to the non-significant difference recorded between coated and mixed fertilisers at each observation time. The comparisons among treatments are presented at each harvest except for percentage of S derived from the fertiliser and percentage of fertiliser S recovery in pasture tops analysed at 5 harvests in split plot in time to establish time trends.

To analyse for serial correlation between repeated harvests the BMDP-2V computer program was initially used to analyse the data. In this analysis there was no serial correlation observed and, on the advice of a statistician, analysis of variance at each harvest was used (Burr, 1982). Because of the complexity of the experimental design, data from the $S \times P \times L$ interaction is generally presented to highlight consistent trends.

Treatment	Treatment		P application
	code	kg ha ⁻¹	mg product pot -1
S source			
Elemental S	E	20	40
Gypsum	G	20	216
P source			
Triple superphosphate	TSP	40	402
Rock phosphate	RP	40	574
S and P application			
S-coated TSP or RP	SC		
S mixed with TSP or RP	SM		
Water management			
Non-leached	NL		
Leached	L		
Control	С		

	Table 4.2	Treatments and treatment codes for pasture ex	periments.
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	Treatment	S	and P appli	cation
Treatment	code	S	P (kg ha ⁻¹)	product (mg pot ⁻¹)
Control	С	0	0	0
TSP + gypsum	G	20	40	618
TSPS-PVA	PVA	20	40	442
TSPS-sodium lig. sul.	LS	20	40	442
TSPS-slack wax	SW	20	40	442
TSPS-hifert	HF	20	40	442

Table 4.3	Source and rate of S and I	P applied under non-leached conditions.

d. Experimental Procedure

Two weeks after the application of basal nutrients and ³⁵S, the treatments shown in Table 4.2 and 4.3 were applied. The fertilisers were spread on the surface of the sward and the pots were watered daily. In the leached treatments leachates were collected and weighed every two days and stored in plastic bottles in a cold room at 4°C. The S and P content of leachates, as well as the radioactivity was measured following each harvest, using ICP Spectrometry and liquid scintillation counting respectively.

Successive harvests were made at intervals of between four and eight weeks depending on the growth rate of the pastures. At harvesting, plants were cut approximately 2 cm above the soil surface and the tops were separated into the grass and clover and oven-dried at 80 °C until a constant weight was achieved. The samples were then ground to pass a 2 mm sieve and digested in a sealed container with a mixture of 70 % perchloric acid and hydrogen peroxide (Anderson and Henderson, 1986). The concentration of S and P in the plant digest was then determined by ICP, and for radioactivity by liquid scintillation counting.

After the fourteenth harvest (96 weeks) the pastures were first cut in the same way as previously. The plant material between 0 and 2 cm was then cut and washed and mixed with the plant top. The soils were then pushed out of the pot and roots were removed. The soils were then mixed thoroughly and air-dried for analysis. Soil analysis included total S (Till *et al.*, 1984) and Ca(H₂PO₄)₂ extractable S (Barrow, 1967). The percentage of S in plant derived from the fertiliser was calculated by the same method as in the previous experiment.

4.3 RESULTS - S AND P SOURCES EXPERIMENT

4.3.1 Yield

a. Clover

For ease of comparison, data of S source (S) \times P source (P) \times leaching (L) interaction are presented in each graph, mainly because of the non-significant differences recorded between coated and mixed fertilisers throughout the experiment and the fact that significant interaction was recorded only among the S, P and L factors, although the interaction between these factors was not always significant at each harvest.

There were no significant differences recorded between leached and non-leached treatments up to the 8th week of application. In harvests between weeks 8 and 44, the combination of gypsum and TSP resulted in significantly lower yield under leached than under non-leached conditions. There was no significant difference in dry matter yield recorded between leached and non-leached treatments in the elemental S treatments at any harvest, although the yield was always lower under the leached condition (Figure 4.1).

There were significant $S \times P$ and $S \times L$ interactions recorded for clover dry matter yield, although these interactions were not significant at each harvest (Appendix 4.1). At all harvests up to week 28 clover yield was higher in the TSP than the RP treatments (Figure 4.1).

Clover yield from the gypsum treatments increased between 4 and 8 weeks and then declined with time and the yield from elemental S increased up to week 17 and declined after this harvest. In the TSP treatments, gypsum resulted in a significantly higher yield than that of elemental S in the first 8 weeks after fertiliser application. At week 12 and under non-leached condition, no significant differences were recorded between gypsum and elemental S treatments. A higher yield with elemental S than with gypsum was recorded in harvests between weeks 17 and 44 (Figure 4.1).

Among the RP treatments, gypsum resulted in significantly higher yield than that of elemental S in the first 8 weeks. However, a higher yield from elemental S was observed at week 28 and in harvests between weeks 44 and 60. In the gypsum treatments, a significantly lower yield was recorded from leached than non-leached treatments in harvests between weeks 24 and 44. There was no significant difference in dry matter yield recorded between the leached and non-leached treatments where elemental S was applied (Figure 4.1).

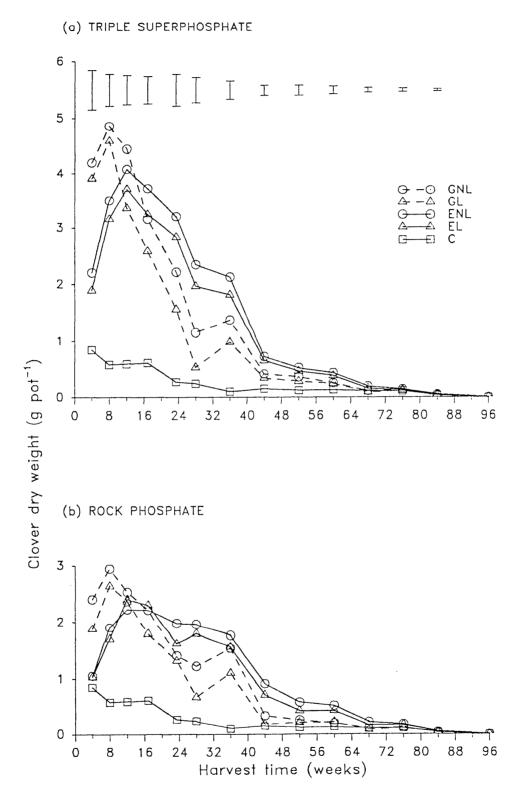
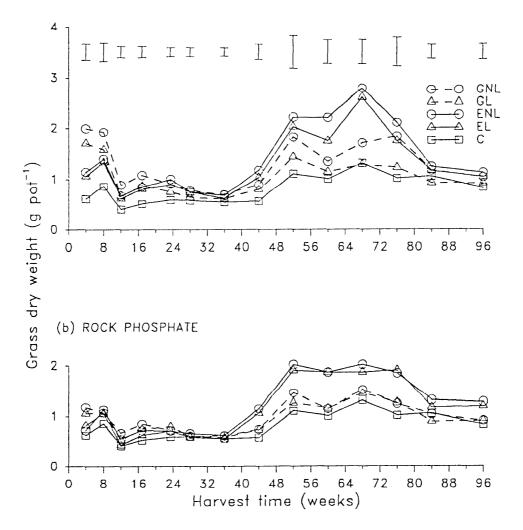


Figure 4.1 The effect of different S and P sources on dry weight of clover under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for $P \times S \times L$ interaction at each harvest.

b. Grass

There were significant $S \times P$ and $S \times L$ interactions recorded in grass dry matter yield, although these interactions were not significant at each harvest (Appendix 4.2). At each harvest, there was no significant difference in grass dry matter yield recorded between leached and non-leached treatments, although the leached treatments tended to produce lower yield than that of non-leached treatments. In general, grass yield in the TSP treatments was higher than in the RP treatments at 8 weeks and no differences were observed after this harvest (Figure 4.2).



(a) TRIPLE SUPERPHOSPHATE

Figure 4.2 The effect of different S and P sources on dry weight of grass under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for S \times P \times L interaction at each harvest.

In general, with gypsum grass yield declined up to week 12 and increased after week 44. The yield with elemental S was relatively constant up to week 44 and increased after this time. The grass response to applied fertilisers was not as strong as that of clover. Among the TSP treatments, gypsum resulted in the highest dry matter yield at 8 weeks. No significant differences were recorded between elemental S and gypsum in harvests between weeks 8 and 52. A significantly higher dry matter yield with elemental S than with gypsum was recorded between weeks 52 and 76 (Figure 4.2).

Among the RP treatments, gypsum resulted in a higher yield than those of elemental S at 4 weeks after application. A significantly higher yield with elemental S than with gypsum was observed in harvests between weeks 44 and 84 (Figure 4.2).

c. Total tops

There were significant $S \times P$ and $S \times L$ interactions recorded in total tops dry matter yield, although the significant interaction was not recorded at each harvest (Appendix 4.3). In general the pasture yield with gypsum declined with time. The yield with elemental S increased up to week 24 and declined after this time. Among the TSP treatments, gypsum resulted in the highest yield at 8 weeks. No differences were recorded between gypsum and elemental S in harvests between weeks 8 and 24. From week 24 up to week 68 higher yields were observed with elemental S than with gypsum, although the yield with gypsum under non-leached conditions did not differ significantly from that of elemental S under leached conditions at weeks 24 and 52. The yield with gypsum was lower under leached than under non-leached conditions in harvests between weeks 8 and 44. No significant differences were observed between the leached and non-leached treatments in the elemental S treatments (Figure 4.3).

Among the RP treatments, gypsum resulted in the highest yield at the first 8 weeks. A significantly higher yield was recorded with elemental S than with gypsum in harvests between weeks 36 and 84. No significant differences were recorded between leached and non-leached treatments, except in the gypsum treatments at weeks 28 and 36. At these harvests there was a lower yield from the leached than from the non-leached treatments (Figure 4.3).

In general, significantly higher dry matter yields were recorded with TSP than with RP in the earlier harvests and no differences were observed after week 44 (Figure 4.3).

d. Roots

There was no significant interaction recorded in root dry matter yield. Highest yield was observed in the combination of elemental S with TSP under non-leached condition, although this did not differ significantly from that under leached conditions. The combination of gypsum with TSP resulted in similar yield to that of the RP treatments (Appendix 4.4).

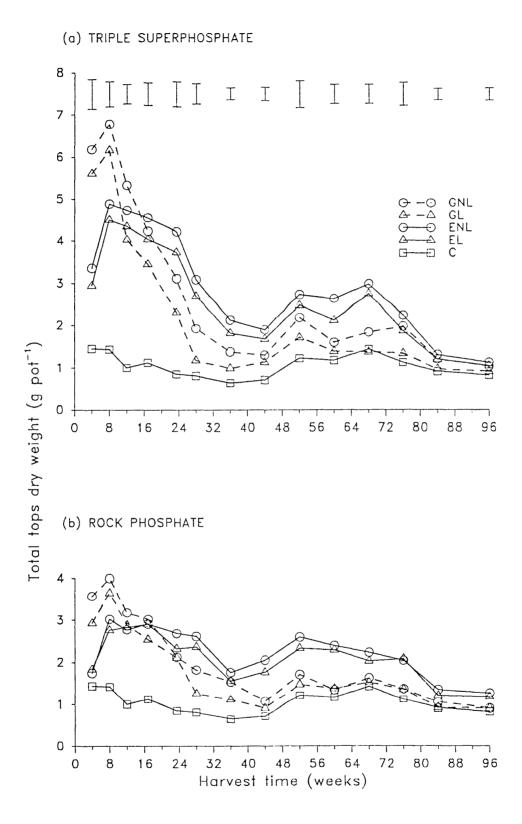


Figure 4.3 The effect of different S and P sources on total tops dry weight under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for S \times P \times L interaction at each harvest.

4.3.2 S Content

a. Clover

There were significant $S \times P$ and $S \times L$ interactions recorded in clover S content, although the interaction was not significant at each harvest (Appendix 4.5). In general, the clover S content with gypsum declined with time. The S content with elemental S increased up to week 12 and then declined. Among the TSP treatments highest S content was recorded in the gypsum treatments at 8 weeks. A significantly higher S content was recorded with elemental S than with gypsum in harvests between weeks 12 and 60. No significant differences were observed after the week 60 harvest (Appendix 4.6).

Among the RP treatments, gypsum resulted in the highest total S content at the first two harvests. This was reversed in harvests between weeks 17 and 60. A significantly lower S content from the leached than the non-leached treatments was recorded in the combination of gypsum with TSP in the first 12 weeks and at week 24 as well as week 36 after fertiliser application. No significant differences were observed between leached and non-leached treatments from the elemental S treatments. In general, a significantly higher S content was recorded with TSP than with RP up to week 24 and no differences were observed after this harvest (Appendix 4.6).

b. Grass

In general, there was no significant interaction recorded in grass S content, except at weeks 44 and 68, where significant $S \times L$ and $S \times P$ interactions were recorded respectively (Appendix 4.7). In general, the grass S content declined with gypsum up to week 12 and increased after week 44. The S content with elemental S was relatively constant up to week 44 and increased after this time. Among the TSP treatments gypsum resulted in the highest S content in the first 12 weeks. A higher S content was recorded with elemental S than with gypsum in harvests between weeks 36 and 76. No differences were observed after week 76 (Appendix 4.8).

Among the RP treatments, no significant differences were recorded between gypsum and elemental S treatments in the first 36 weeks. A higher S content was recorded with elemental S than with gypsum in harvests between weeks 36 and 76 (Appendix 4.8). In general, no significant differences were recorded in grass S content between TSP and RP treatments. Leaching had no significant effect on grass S content, except with gypsum and TSP at week 12.

At this harvest a significantly lower S content was recorded in the leached than in the nonleached treatments (Appendix 4.8).

c. Total tops

There were significant $S \times P$ and $S \times L$ interactions recorded in total tops S content (Appendix 4.9). In general the total tops S content declined with time in the gypsum treatments. The S content with elemental S increased up to week 17 and declined after this time. Among the TSP treatments, gypsum resulted in the highest S content at 8 weeks. A significantly higher S content was recorded with elemental S than with gypsum in harvests between weeks 12 and 76. No differences were observed after week 76. Under leached conditions the combination of gypsum and TSP resulted in a significantly lower S content than that of non-leached conditions in the first 12 weeks and at week 36. No significant differences were observed between the leached and non-leached treatments in the elemental S treatments either with TSP or RP (Figure 4.4).

Among the RP treatments, gypsum resulted in a significantly higher S content than that of elemental S at the first two harvests. This was reversed in harvests between weeks 17 and 76. No significant differences were recorded between leached and non-leached treatments at each harvest, although a lower rate was observed in the gypsum treatment under leached than under non-leached conditions at the first harvest (Figure 4.4). In general, the S content was higher with TSP than with RP in earlier harvests and no significant differences were recorded after week 24 (Figure 4.4).

d. Roots

There were no significant differences between treatments in root S content, although the S content with gypsum under leached conditions tended to be lower than that of elemental S under non-leached condition (Appendix 4.4).

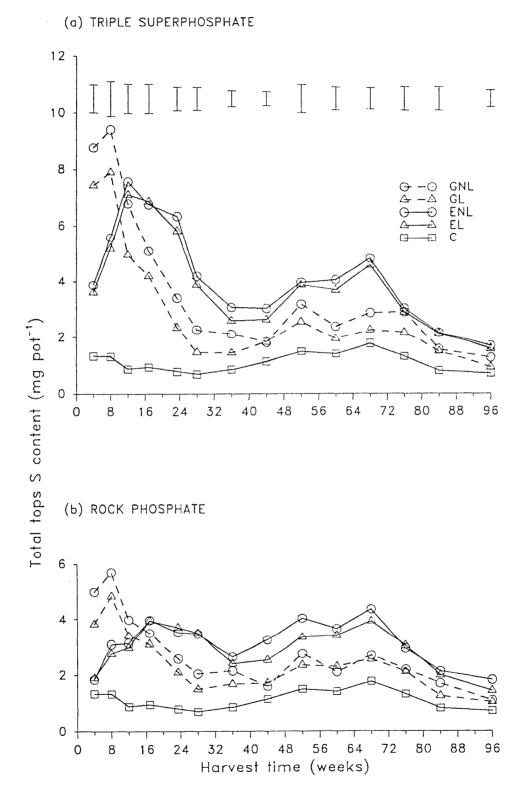


Figure 4.4 The effect of different S and P sources on total tops S content under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for S \times P \times L interaction at each harvest.

4.3.3 P content

a. Total tops

At weeks 36 and 68, there was a significant $S \times P$ interaction recorded in pasture P content (Appendix 4.10). Among the TSP treatments, gypsum resulted in the highest P content up to week 17. In harvests between weeks 17 and 36 and between weeks 36 and 68, a significantly higher P content was recorded with elemental S than with gypsum. Under leached conditions, the gypsum treatment resulted in significantly lower P content than that of under non-leached conditions between weeks 36 and 68. No significant differences were recorded in harvests between weeks 68 and 96, except in the gypsum treatments under leached conditions, which was lower than that of elemental S under non-leached conditions (Table 4.4).

Among the RP treatments, gypsum resulted in significantly higher P content than elemental S in the first 4 weeks. This was reversed in harvests after week 36. The highest total P content was recorded in the elemental S + TSP treatments, although this did not differ significantly from that of gypsum + TSP (Table 4.4).

In general, a higher P content was recorded with TSP than with RPin the first 36 weeks and no significant differences were observed after week 36 (Table 4.4).

Treatment			intent (mg pot ⁻¹) ïmes (weeks)		
	0 - 17	17 - 36	36 - 68	68 - 96	Total
T S P ENL EL GNL GL	40.39 b ^A 37.62 b 47.99 a 44.59 a	19.57 a 18.61 a 14.38 bc 13.32 cd	15.53 a 14.52 ab 10.51 c 8.65 d	6.94 ab 6.59 a-c 5.47 b-d 5.12 cd	82.43 a 77.34 b 78.35 ab 71.68 c
R P ENL EL GNL GL	21.15 d 20.46 d 25.91 c 24.77 c	14.87 bc 15.81 b 12.92 cd 11.48 d	15.17 ab 13.65 b 8.68 d 8.44 d	7.68 a 6.79 ab 5.16 cd 4.55 d	58.87 d 56.71 d 52.67 d 49.24 e

 Table 4.4 The effects of S and P sources on P content of pastures under leached and nonleached conditions.

A Data followed by the same letter in the same column are not significantly different (P > 0.05) (Duncan's multiple range test)

b. Roots

In general, the root P content did not differ significantly among the treatments, except in the gypsum treatments, which resulted in a significantly lower P content under leached than under non-leached conditions (Appendix 4.4).

4.3.4 Percentage of S derived from the fertiliser

a. Tops

There was significant $S \times P$ interaction recorded in the percentage of S in pastures derived from the fertiliser, although the significant interaction was not recorded at each harvest (Appendix 4.11). In the gypsum treatments, the percentage of S derived from the fertiliser declined significantly over time. However, in the elemental S, TSP combination, the rate increased significantly up to week 8 and declined after that time. In the elemental S, RP combination the percentage of S derived from the fertiliser increased significantly up to week 3 and declined after that time. In the elemental S, RP combination the percentage of S derived from the fertiliser increased significantly up to week 36 and declined after that time (Table 4.5).

In general, gypsum treatments had a higher percentage of S in the tops derived from the fertiliser than did elemental S at the first two harvests. This was reversed in harvests between weeks 8 and 96. Leaching had no significant effect on the percentage of S in the plant derived from the fertiliser, except in the gypsum, RP combination at week 36 where a significantly lower proportion of S was derived from the fertiliser in the leached than the non-leached treatment (Table 4.5).

Treatment		S derived fr	om the fertiliser t Times (weeks)	(%)	
	4	8	36	68	96
T S P ENL EL GNL GL	27.8 j ^A 27.6 j 77.7 a 76.3 ab	50.5 e 49.8 e 72.9 bc 71.4 c	43.7 f 41.3 fg 32.0 hi 29.0 ij	18.0 k 17.3 k 10.7 l 8.9 lm	8.0 l-n 8.3 l-n 7.0 mn 5.7 n
R P ENL EL GNL GL	19.4 k 18.9 k 72.1 bc 70.3 c	27.1 j 29.1 ij 65.9 d 64.8 d	51.6 e 50.4 e 38.0 g 33.1 h	20.2 k 16.7 k 10.2 lm 9.9 lm	9.2 lm 9.5 lm 5.8 n 5.5 n

 Table 4.5 The effect of S and P sources on the percentage of S in the tops derived from the fertiliser under leached and non-leached conditions.

^A Data followed by the same letter are not significantly different (P > 0.05) (Duncan's multiple range test).

b. Roots

Elemental S treatments had the highest percentage of S in root derived from the fertiliser and no significant differences were recorded between leached and non-leached treatments or between TSP and RP (Appendix 4.4).

4.3.5 Recovery of fertiliser S

a. Total tops

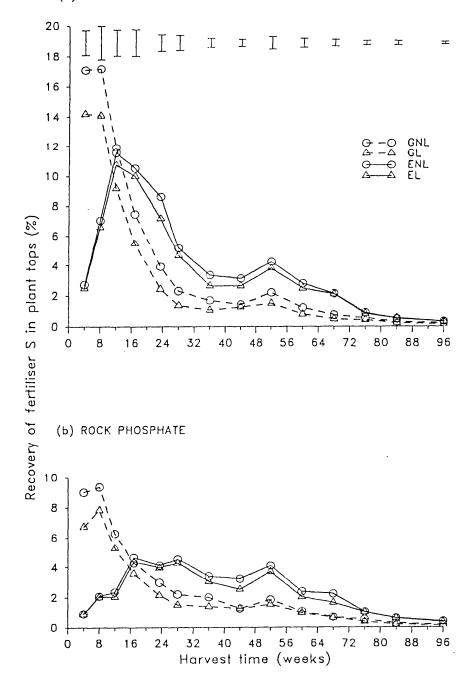
There were significant $S \times P$ and $S \times L$ interactions recorded in the recovery of fertiliser S in the tops, although there was no significant interaction recorded after week 28 (Appendix 4.12). Among the TSP treatments, gypsum resulted in significantly higher recovery than that of elemental S at the first two harvests. This was reversed in harvests between weeks 12 and 76 (Figure 4.5). A significantly lower recovery of fertiliser S was recorded with gypsum in the leached than in the non-leached treatments in the first 24 weeks. No significant differences were recorded between the leached and non-leached treatments with elemental S, except at weeks 24 and 36 where the leached treatment resulted in a lower recovery (Fig 4.5).

Among the RP treatments, the recovery of fertiliser S in the tops was higher with gypsum than with elemental S at 12 weeks. This was reversed in harvests between weeks 24 and 76 (Figure 4.5). No significant differences were observed between the leached and non-leached treatments at each harvest, although the recovery of fertiliser S in the gypsum treatment was significantly higher in the non-leached than in the leached treatment at week 4 (Figure 4.5).

In general, the recovery of fertiliser S in the TSP treatments was significantly higher than that in RP treatments at the first five harvests and no significant differences were observed after week 24 (Figure 4.5).

b. Roots

In general, the recovery of fertiliser S in roots was significantly higher with elemental S than with gypsum and no significant differences were observed between leached and non-leached treatments (Appendix 4.4).



(a) TRIPLE SUPERPHOSPHATE

Figure 4.5 The effect of different S and P sources on the recovery of fertiliser S in plant tops under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for $S \times P \times L$ interaction at each harvest.

4.3.6 S and P loss through leaching

a. S content of leachates

At each harvest, there was no significant interaction recorded in the amount of S in leachates among the treatments (Appendix 4.13). Up to week 36 the highest S content of leachates was recorded in the gypsum treatments, and the loss from the RP treatments was lower in the first 17 weeks. No significant differences were observed between the treatments in harvests between weeks 36 and 68 and 68 and 96. The total S loss was lower with elemental S than with gypsum (Appendix 4.14).

b. P content of leachates

There was no significant interaction recorded in the P content of leachate at each observation (Appendix 4.15). In general, the P content of leachates did not differ significantly among the treatments, although it was higher from TSP than from RP in the first 17 weeks (Appendix 4.16).

c. Percentage of S in leachates derived from the fertiliser

There were significant $S \times P$ interactions in the percentage of S in leachates derived from the fertiliser at week 4 and 68 (Appendix 4.17) with the level generally declining with time with the gypsum and increasing up to week 36 and then declining in the elemental S treatments (Table 4.6).

Treatment			om the fertiliser (° t Times (weeks) -	· ·	
	4	8	36	68	96
TSP E G	30.87 f-h ^A 92.94 a	70.40 c 83.79 b	66.58 c 58.70 d	37.79 e 36.76 ef	26.73 gh 24.91 h
R P E G	9.96 i 92.05 a	67.54 c 81.04 b	65.01 cd 59.67 d	38.23 e 31.89 e-g	26.27 gh 24.41 h

 Table 4.6 The effect of S and P sources on the percentage of S in leachates derived from the fertiliser.

A Data followed by the same letter are not significantly different (P > 0.05) (Duncan's multiple range test)

d. Recovery of fertiliser S in leachates

There were significant $S \times P$ interactions recorded in the amount of fertiliser S recovered in the leachates at week 4 and 12 (Appendix 4.18). In general, fertiliser S losses were higher from the gypsum treatments in the first 17 weeks. No significant differences were recorded after week 17 (Figure 4.6).

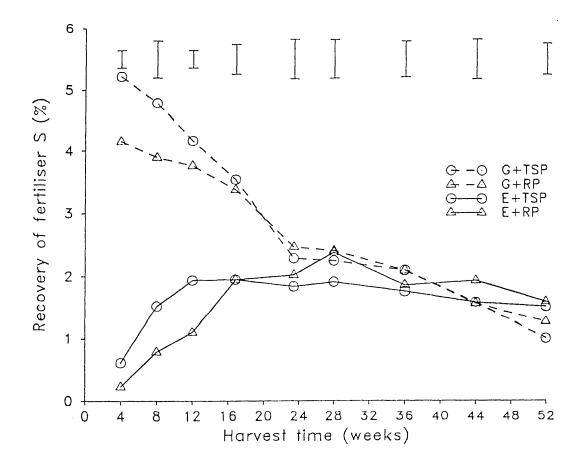


Figure 4.6 The effect of different S and P sources on the recovery of fertiliser S in leachates. Vertical bars indicate LSD (P = 0.05) for S × P interaction at each observation time.

4.3.7 Soll S pools

At the end of the experiment, the extractable S levels did not differ significantly among the treatments. No significant differences were observed in the percentage of S derived from the fertiliser and in the recovery of fertiliser S in the extractable S fraction among the treatments (Appendix 4.19).

There was no significant difference in organic S level recorded between the treatments (Appendix 4.19). However, significant $S \times P$, $S \times L$ and $S \times P \times L$ interactions were recorded in the percentage of S in this fraction derived from the fertiliser. In general, the percentage of S derived from the fertiliser in the organic S fraction was higher in the RP than in the TSP treatments (Appendix 4.19).

At the end of the experiment, there were no significant differences in soil total P content recorded among the treatments (Appendix 4.20).

4.4 RESULTS - S SOURCES EXPERIMENT

4.4.1 Yield

For ease of presentation of data, two or more harvests have been combined. At weeks 4 + 8, highest yield was recorded in the G treatment, intermediate yields with PVA and LS and lowest yield in the control. At weeks 12 + 17, the ranking was G = PVA = LS > SW = HF > C; at weeks 24 + 28, PVA = LS = HF = SW > G > C and at weeks 36 + 44, SW = HF > PVA = LS > G > C. Highest total yield was recorded in the G and lowest in the C treatment (Table 4.7).

4.4.2 S content

Highest S content was recorded in the G treatment at weeks 4 + 8. At these Harvests the S content with PVA was not significantly different from that with LS and this was higher than SW and HF. The lowest S content was in the control at each harvest. At weeks 12 + 17, highest S content was recorded with PVA and LS whilst at 36 + 44 SW and HF had the highest S content. The highest total S content was recorded in the LS treatment, although this did not differ from that of G and PVA. Among the fertilised treatments, SW resulted in the lowest S content, although this was similar to that of HF and G (Table 4.7).

4.4.3 P content

Highest P content was recorded in the G treatment at weeks 4 + 8 with intermediate levels with PVA and LS. The lowest P content was recorded in the C treatment at each observation time (Table 4.7). At weeks 12 + 17, highest P content was recorded in the G, LS and PVA treatments. At weeks 24 + 28 and 36 + 44 the ranking was SW = HF > PVA = LS > G > C. Highest total P content was recorded in the G, LS and PVA treatments (Table 4.7).

Treatment Harvest Times (weeks)						
Treatment			24 + 28		Total	
Yield (g pot	-1)					
G	13.5 a ^A	9.2 a	4.8 b	2.5 c	30.0 a	
PVA SW HF LS	8.0 b 5.2 c 5.5 c 8.6 b	9.2 a 8.1 b 7.8 b 9.2 a	7.5 a 8.6 a 8.4 a 7.6 a	4.2 b 5.5 a 5.6 a 3.6 b	28.9 ab 27.4 b 27.3 b 29.0 ab	
С	2.9 d	2.3 c	1.7 c	1.4 d	8.3 c	
S content (mg pot ⁻¹)					
G	18.8 a	11.1 b	5.6 b	3.6 c	39.1 a-c	
PVA SW HF LS	8.6 b 5.8 c 6.1 c 9.8 b	14.2 a 10.7 b 10.7 b 14.1 a	10.9 a 12.2 a 12.2 a 11.0 a	6.5 b 8.1 a 8.3 a 5.8 b	40.2 ab 36.8 c 37.3 bc 40.7 a	
С	2.8 d	2.0 c	1.5 c	1.9 d	8.3 d	
P content (mg pot ⁻¹)					
G	28.1 a	19.8 ab	10.5 c	5.1 c	63.5 ab	
PVA SW HF LS	17.9 b 11.2 c 11.0 c 20.5 b	22.1 a 15.7 c 16.7 bc 23.6 a	15.5 b 18.9 a 18.8 a 16.3 b	9.0 b 13.1 a 14.2 a 9.2 b	64.5 ab 58.9 b 60.6 b 69.6 a	
С	4.2 d	3.4 d	2.5 d	2.2 d	12.3 c	

Table 4.7 The effect of S source on dry weight (g pot ⁻¹),	S and P content (mg pot ⁻¹) of plant
tops at different harvest times.	

A Data in each component followed by the same letter in the same column are not significantly different (P > 0.05) (Duncan's multiple range test).

4.4.4 Percentage of S derived from the fertiliser

In the gypsum treatment, the percentage of S derived from the fertiliser in the plant tops declined significantly with time after week 8 (S source × harvest time interaction). In the PVA and LS treatments, the contribution of S from the fertiliser increased up to week 17 and then declined, whilst in the HF and SW treatments the rate increased up to week 28 and then declined (Table 4.8).

In general, the percentage of S derived from the fertiliser was higher with gypsum than with the elemental S in earlier harvests and this was reversed after week 17 (Table 4.8).

Treatment			om the fertiliser (Times (weeks)		
meatment	4	8	17	28	44
G	78.96 a ^A	73.41 a	57.03 cd	40.13 g	31.28 hi
PVA SW HF LS	27.46 i 17.93 j 18.39 j 27.72 i	49.84 f 32.90 h 31.34 h 50.47 ef	63.37 b 55.34 de 56.62 cd 62.08 bc	51.32 ef 58.45 b-d 57.10 cd 51.48 ef	41.59 g 47.10 f 44.07 f 39.61 g

Table 4.8 The effect of S source on the percentage of S in pastures derived from the	fertiliser at
different harvest times.	

^A Data followed by the same letter are not significantly different (P > 0.05) (Duncan's multiple range test).

4.4.5 Recovery of fertiliser S

Gypsum resulted in the highest recovery of fertiliser S in the plant tops at weeks 4 + 8. No differences were observed between PVA and LS and also between HF and SW (Table 4.9). At weeks 12 + 17, highest recovery was recorded in the PVA and LS treatments, whilst the lowest recovery was recorded from SW and HF. The ranking at weeks 24 + 28 and 36 + 44 was HF = SW > LS = PVA > G. The total recovery of fertiliser S was highest in the gypsum treatment and lowest with SW and HF. A non-significant difference was also recorded between PVA and LS (Table 4.9).

 Table 4.9 The effect of S source on the recovery of fertiliser S (%) in pastures at different harvest times.

Treatment	4 + 8	Harvest 11 12 + 17	mes (weeks) 24 + 28	36 + 44	Total
G	35.7 a ^A	17.8 b	6.0 c	2.9 c	62.4 a
PVA	8.8 b	21.8 a	14.6 b	7.0 b	52.3 b
SW HF	3.9 c 4.1 c	12.1 c 12.7 c	17.6 a 17.8 a	9.9 a 10.1 a	43.5 c 44.7 c
LS	10.0 b	22.0 a	14.7 a	6.1 b	52.9 b

A Data followed by the same letter in the same column are not significantly different (P > 0.05) (Duncan's multiple range test).

4.4.6 Soil S pools

At the end of the experiment, the extractable S levels did not differ significantly among the S sources and no significant differences were observed in the percentage of S in this fraction derived from the fertiliser. The recovery of fertiliser S in this fraction was highest with HF, PVA, SW and LS (Appendix 4.21). There was no significant difference in organic S level recorded among the treatments (Appendix 4.21). The percentage of S derived from the fertiliser and the recovery of fertiliser S in this fraction did not differ significantly among the treatments (Appendix 4.21).

At the end of the experiment, there was no significant difference in total P content recorded among the treatments (Appendix 4.22).

4.5 DISCUSSION

a. S and P sources

In the main experiment, no significant differences were observed between coated and mixed fertilisers at each harvest, although the mixed fertiliser tended to produce higher yield in the early harvests. This was probably because the water soluble glue which was used to bind elemental S with the granulated fertilisers did not impair the oxidation of elemental S.

A rapid reduction in clover growth was recorded and higher grass yields were measured after week 36. This is the result of N transfer from the legume to the grass which has been shown by Walker and Adams (1958). They also demonstrated that grass was a more effective competitor for limited sulfur than white clover.

The lower dry matter yield and S content of the clover in the elemental S treatments in the first two harvests resulted from the delay in oxidation of elemental S. Sulfatic fertiliser such as gypsum has been reported by Scott *et al.*, 1983; Klessa *et al.*, 1989 to be effective in supplying S to pasture in the first year of application with a small residual value observed from this source.

The percentage of S derived from the fertiliser and the recovery of fertiliser S in the plant data indicated different rates of sulfate release from the different sources of S. Gypsum, in which the sulfur is in the soluble sulfate form, resulted in an immediate release of sulfate to the plant as reflected by the higher recovery of fertiliser S in the plants from gypsum at the early harvests. On the other hand, the elemental S provided a slower and more sustained sulfate release. A similar response in an irrigated white clover pasture in South-east Queensland was reported between gypsum and elemental S Bowdler and Pigott (1990). They found that both flowers of sulfur and gypsum were effective as source of S for pastures.

The effectiveness of elemental S in providing S to crops depends on the oxidation rate and this is influenced by many factors including soil temperature (Li and Caldwell, 1966, Nor and Tabatabai, 1977, Janzen and Bettany, 1987b), soil moisture (Janzen and Bettany, 1987b, Nevell and Wainwright, 1987), soil pH (Nor and Tabatabai, 1977), soil organic matter (Wainwright *et al.*, 1986), soil texture (Rehm and Caldwell, 1969), particle size of elemental S (Blair, 1987, Janzen and Bettany, 1987a), sulfur-oxidizing microorganisms (Vitolins and Swaby, 1969) and nutrient status of the soil (Bloomfield, 1967). Activity of sulfur-oxidizing microorganisms in soil is of major importance where elemental S-containing fertilisers are used. Many different microorganisms are reported to be capable of oxidizing inorganic S compounds including chemolithotropic organisms such as the *Thiobacillus* spp. and a variety of heterotrophic bacteria, actinomycetes and fungi (Wainwright *et al.*, 1986). Lindemann *et al.*(1991) found that no oxidation in the elemental S occurred in the absence of *Thiobacillus* spp.

Although there was a significant $S \times P$ interaction in pasture P content (Appendix 4.10), the availability of S in the soil was the most important factor. This was described by the fact that higher P content was recorded in the gypsum treatment up to week 17. However, between weeks 17 and 68 a higher P content was recorded in the elemental S treatment (Table 4.4). This was most likely due to the higher availability of S as a result of S oxidation.

The higher yield with TSP was due to the higher availability of P from this source compared to RP, since the availability of P from RP is dependent on the dissolution rate of this material. This result is consistent with the previous results reported by Bolland *et al.* (1988). Rajan (1987) reported that the reactive phosphate rocks applied with or without sulfur were as effective as superphosphate in the first and third year of application when used in soils of pH less than 5. The pH of the soil used in the present experiment was 6.1 (1:5 H₂O); therefore rockphosphate was not an effective source of P in this pH range. This was compounded by the granulation of the RP source.

It is generally accepted that the phosphate rock is effective when it is finely ground, reactive and applied to acid soils of low exchangeable calcium (Khasawneh and Doll, 1978). Many factors are associated with the dissolution of RP, including the source of rock : reactive rock phosphate is more effective than non-reactive (Hughes and Gilkes, 1986a); soil acidity (Hughes and Gilkes, 1986b, Kanabo and Gilkes, 1987, Rajan *et al.*, 1991 a, b); particle size (Khasawneh and Doll, 1978, Kanabo and Gilkes, 1988b, Gilbert *et al.*, 1990); rate of application (Bolland and Barrow, 1988) and method of application (Kanabo and Gilkes, 1988a). Finer particle size increases the agronomic effectiveness of RP because of the increase in surface area which leads to an increase in the degree of contact between RP and soil (Olsen, 1975). The effectiveness also depends on the degree of mixing of RP with soil, with maximum effectiveness when it is uniformly mixed (Khasawneh and Doll, 1978). In the present study, regranulation of RP probably reduced its effectiveness due to the reduction of the surface area.

Although many studies have found that RP was an ineffective source of P in the year of application (Chien and Hammond, 1978 and Bolland and Gilkes, 1990), a greater residual value of this material has been observed compared with single superphosphate (Mackay and Wewala, 1990).

In the leaching treatments, only small amounts of P were detected in the leachates during the course of the experiment. This would most likely be associated with the low mobility of P in the soil due to adsorption. Direct extrapolation of results from pot experiments to the field situation is dangerous. However, the loss of 2.37 mg P pot⁻¹ (Appendix 4.16) from TSP equates to 1.3 kg ha⁻¹. This agrees with other studies reported in the literature (Brady, 1990). The movement of P within the soil profile is affected by soil pH, its concentration in the soil solution and the nature of the adsorption complex in the soil (Bar-Yosef *et al.*, 1988). A leaching-loss study of nitrogen, phosphorus and potassium was conducted recently on tobacco soils in North Queensland (Littlemore *et al.*, 1990). They estimated that 50 - 80 kg P ha⁻¹ was lost through leaching. Similar results were reported by Weaver et al. (1988 a, b).

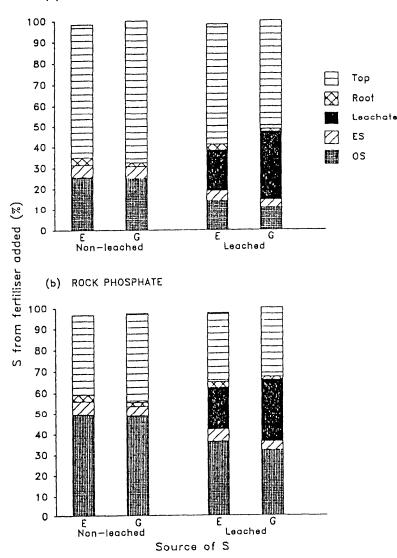
Under leached conditions higher clover yield was recorded in the gypsum than in the elemental S treatments in the early harvests. However, after week 8 clover yield and S content were lower in the gypsum than the elemental S treatments. This was because of the combination of higher S offtake and greater loss of S through leaching when gypsum was applied. The low loss of fertiliser S from elemental S is consistent with the results of Jones *et al.*, 1968 and Korentajer *et al.*, 1984. The movement of sulphate in the soil is influenced by the amount of water movement within the soil, the particle size of the product used (Korentajer *et al.*, 1984), its concentration in the soil solution, and its reaction with solid phase components of the soil (Harward and Reisenauer, 1966).

Although there was no significant difference in dry matter yield between leaching and non-leaching in the elemental S treatments, the yield under leaching conditions was always lower than non-leaching conditions. This was probably due to the reduced aeration, from the reduction in pore space associated with the watering regime in the leached treatments, which resulted in slower oxidation of elemental S.

S loss through leaching was also affected by the P sources. In this study the recovery of S in the leachate was higher from the TSP than from the RP treatments in the early harvests (Appendix 4. 14). The most likely reason for this is through competition by phosphorus for adsorption sites in the soil (Parfitt, 1982 and Barrow, 1970). The sulfate desorbed is easily lost through leaching or taken up by the plant. Liming has also been reported to increase S losses

through leaching and generally has a stronger effect than phosphate in relation to S leaching (Chao *et al.*, 1962a).

Figure 4.7 shows the fate of fertiliser S and its transformation into the different soil S and plant pools. This figure illustrates that in the RP treatments a higher amount of applied S was recovered in to the soil organic S pool due to the poorer growth of pastures which resulted in less S uptake by the plant. A higher S loss through leaching with gypsum than with elemental S was also recorded, which is similar to the previous findings of Mahler and Maples (1987).



(a) TRIPLE SUPERPHOSPHATE

Figure 4.7 The effect of S and P sources on the percentage of fertiliser S incorporated into the plant and soil components under leached and non-leached conditions.

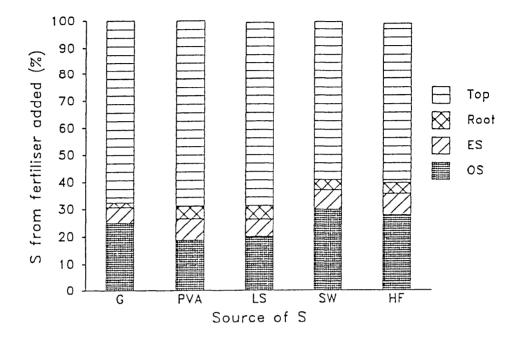
b. Comparison of S sources

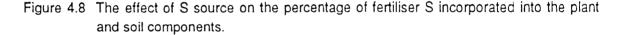
Whilst the effectiveness of different sources of S in a wide variety of crops has been investigated (Gupta and MacLeod, 1984, Dijksterhuis and Oenema, 1990 and Lindemann *et al.*, 1991), there are few studies which have examined TSP-S combinations. In this study, gypsum treatments resulted in the highest yields in the early harvests; however, between weeks 8 and 24, no differences were observed in the pasture yield between gypsum and PVA or LS treatments (Table 4.7).

A lower yield in the SW and HF than in the PVA and LS treatments in the early harvests is most likely due to the impairment of S oxidation from these materials because of the slow dispersion of elemental S from the granules. The use of slack wax to bind elemental S to the TSP granules in this study resulted in the impairment of water penetration into the granules which inhibited elemental S dispersion in the soil. The use of wax in coated or cogranulated commercial fertilisers is generally aimed at slowing nutrient release from the product (Beaton and Fox, 1971).

Chapman (1989) reported that additives (wetting and diffusing agents) in commercial elemental S sources inhibited oxidation of elemental S at high concentration but stimulated it at low concentration. In this experiment the different adhesives clearly showed a different response, particularly early after fertiliser application. PVA and LS, which are soluble in water, released more S compared to SW and HF.

Figure 4.8 shows the fate of S from the different sources. This figure illustrates that there were no significant differences in total recovery of fertiliser S in the pasture tops among the gypsum, PVA and LS treatments. The recovery from HF and SW was similar and was significantly lower than that of gypsum. There were no significant differences in the recovery of fertiliser S in roots among the treatments. A higher amount of S was recovered in the organic S pool from HF and SW than from gypsum, PVA and LS. This most likely results from the slower S release from HF and SW products which resulted in poorer pastures growth. Immobilization of S which was released from these products is probably the main reason for higher S transformation into the organic S fraction.





Using the model developed by McCaskill and Blair (1989), PVA and LS are more appropriate S-coated fertilisers to match the S demand for pastures compared to SW and HF, hence it was observed that there was a significant correlation between predicted S demand and recovery of fertiliser S observed in the pasture tops with a stronger correlation recorded in the PVA and LS compared to HF and SW (Figure 4.9).

This study has shown that gypsum, PVA and LS were effective S Sources for pastures. However, in areas with high rainfall and low S adsorption capacity PVA or LS could be more appropriate because of less S loss by leaching. In addition, the slower release of S from the elemental source in these products avoids the extremely high S concentration found in gypsum fertilised pastures following S application and hence improves the efficiency of S cycling.

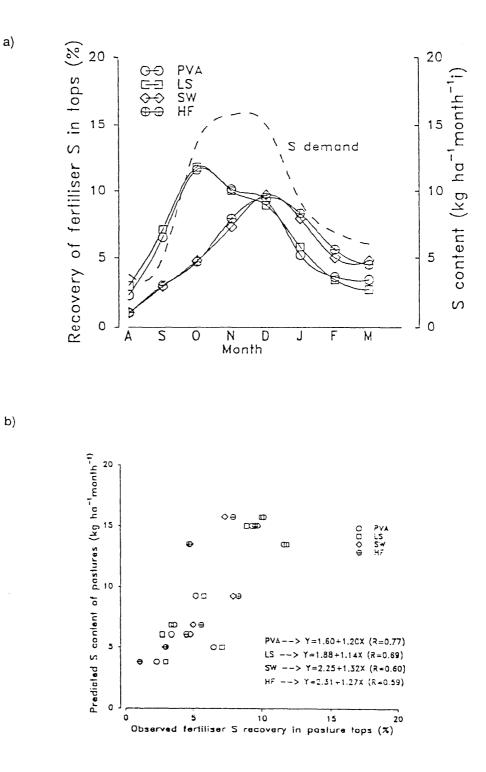


Figure 4.9 a) Prediction of pasture S content and observed fertiliser S recovery in pasture tops and b) correlation between predicted S content and recovery of fertiliser S in pasture tops as influenced by different S sources.

CHAPTER 5

PHYSICAL TESTS OF S-COATED FERTILISERS

5.1 INTRODUCTION

In the experiments reported in this thesis, coated fertilisers were tested under flooded and non-flooded rice and under pasture conditions and showed different S release patterns. The response of crops to different coated fertilisers appears to be related not only to the chemical characteristics but also to physical properties of the fertilisers.

Some measurements which are commonly undertaken to determine physical properties of fertilisers include hygroscopicity, hardness and caking tendency (Bhatti, 1991). Studies on the factors influencing the rate of nutrient release from coated fertilisers and the evaluation of adhesive materials on nutrient release have mainly focused on nitrogen and phosphorus fertilisers (Oertli and Lunt, 1962; Dahnke *et al.*, 1963; Brown *et al.*, 1966; Hall and Baker, 1967; Giordano and Mortveld, 1970).

Two physical characteristics need to be considered in choosing coated fertilisers in order to achieve the maximum efficiency of fertiliser application. The first is the strength of bonding of the coat against frictional forces. This is an important consideration during transportation and application. The second is the strength under the conditions experienced in or on the soil. This relates directly to the agronomic effectiveness of the products. The rate of dispersion of the coated fertiliser under soil conditions directly affects the rate at which the fertiliser comes in contact with plant roots.

In this study, two experiments were undertaken to investigate the physical strength of coated fertilisers against friction forces and against simulated rain.

5.2 EXPERIMENT 1 - MECHANICAL TEST

5.2.1 Materials and methods

A 30 cm diameter and 4 cm deep perspex cylinder ("friabilator") was set up to rotate at 25 rpm on its central axis, in a vertical plane (Scott, 1986). During each rotation the material placed in the friabilator was lifted by a small plate attached to the edge of the cylinder, dropped from a

height of approximately 15 cm and then rolled for approximately 3/4 of the circumference until the plate was encountered again.

Prior to the friabilator test, three sub samples (0.002 g) of intact granules of each coated fertiliser as described in Chapters 3 and 4 : TSP-S with PVA (PVA), TSP-S with sodium lignosulfonate (LS), TSP-S with slack wax (SW) and TSP-S Hifert (HF) were digested separately for total S and P using a nitric-perchloric digestion mixture in which potassium dichromate was included (Till *et al.*, 1984). The S and P content of the fertiliser extracts were determined by ICP.

Three samples (20 g) of each fertiliser were placed separately in the friabilator. Rotation was stopped after 1, 2, 4, 8, 16 and 32 minutes. At each of these times the material was sieved through a 20 mesh (841 μ m) screen. The fine material was removed and weighed, and the coarse material was returned for further testing. A sub-sample (0.001 g) of the fine particle was taken and then digested for total S and P using the same procedure as previously. The results were subjected to analysis of variance as a split plot in time using the same computer program outlined in Chapter 3.

5.2.2 Results and discussion

The S and P contents of the intact granules were not different among the coated fertilisers tested. The S content ranged between 10.6 (LS) to 10.9 % (SW) and the P content ranged between 19.6 (LS) to 20.4 % (HF).

There was no phosphorus detected in the fine particles after the coated fertilisers were tested in the friabilator. This indicated that the fine particle lost from the granules was derived from the coat of the granules.

There was a significant fertiliser source \times time interaction recorded in the amount of particulate loss from the granules. The amount of particle loss increased with time in all treatments, but the rate and extent of loss differed. The highest particle loss was recorded in the HF and there were no significant differences recorded among the PVA, LS and SW (Figure 5.1).

There was a significant fertiliser source \times time interaction recorded in the amount of elemental-S lost from the granules. The amount of loss increased with time in all treatments with the highest loss recorded in the HF with no significant differences recorded among the PVA, LS and SW (Figure 5.2).

In interpreting these findings, the history of the products needs to be considered, for instance, the length of storage time, storage conditions or handling method during

CHAPTER 5 : PHYSICAL TESTS OF S-COATED FERTILISERS

transportation. Storage and bagging are important stages in the fertiliser manufacture before the fertiliser is ready to be used by consumers (Tisdale and Nelson, 1975). The HF material was obtained from Hi-Fert Pty. Ltd. and the storage length was not known, whereas the PVA, LS and SW products were produced in a bench plant and had not been subjected to transportation.

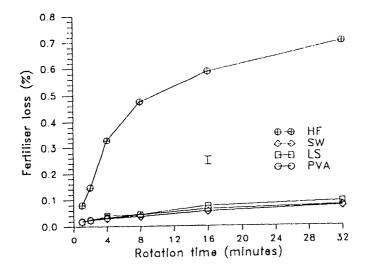


Figure 5.1 Effect of fertiliser source and rotation time on fertiliser loss (%) during the rotation period. Vertical bar indicates the 5% LSD for the fertiliser source × rotation time interaction.

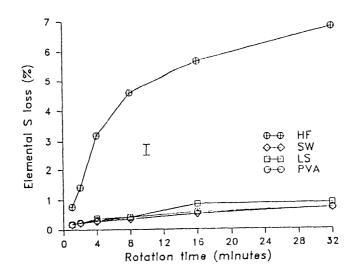


Figure 5.2 Effect of fertiliser source and rotation time on elemental S loss (%) during the rotation period. Vertical bar indicates the 5% LSD for the fertiliser source × rotation time interaction.

5.3 EXPERIMENT 2 - LEACHING EXPERIMENT

5.3.1 Materials and methods

Two gram samples of each fertiliser, TSP-S with PVA (PVA), TSP-S with sodium lignosulfonate (LS) and TSP-S Hifert (HF) were placed in a 3.4 cm (internal diameter) and 7.5 cm (high) plastic vial between layers of 500 μ m nylon mesh. Droplets of deionized water were pumped onto the fertiliser at a rate of 20 mL min⁻¹ through three 1.5 mm internal diameter tubes. The solution and fine material ran to waste. The fertiliser remaining between the layers of mesh was collected, after 3, 6, 12, 24, 36, 48 and 96 hours, dried at 30 °C, weighed and analysed for calcium and phosphorus content after digestion in a mixture of perchloric and nitric acid (Till *et al.*, 1984) and also for elemental S remaining in the granules after extraction with acetone (Shedley, 1982). The experiment was replicated three times. The same statistical procedure as used in the physical strength study was used to analyse the data.

Before and after 24 hours of leaching, one granule was taken from each fertiliser. These granules were dried in an oven at 80 °C for 4 hours. The granule was mounted on a bronze stub (0.5 cm diameter) with Selleys superglue and rubbed with a fine sandpaper to form a cross-section. The granule was then gold-coated and a photograph of the cross-section taken under an electron microscope. A cross-section of each fertiliser was also scanned using the electron microprobe. The microscopic and scanning work was conducted by Mr. Rick Porter, Electron Microscope Unit, University of New England. The scanning for S was conducted on four spots of the granule surface as shown in Figure 5.3.

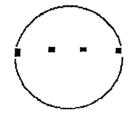


Figure 5.3 Cross-section of S coated fertiliser and areas where the scanning microprobe was used.

A separate test was also conducted to study the dispersion of different coated fertilisers in distilled water at different times (0, 48 and 96 hours). In this study, one granule of each product : TSP-S with PVA (PVA), TSP-S with slack wax (SW), TSP-S Hifert (HF) and TSP-S with sodium lignosulfonate (LS), was placed in a petridish (4 cm diameter) which was filled with distilled water. The petridishes were left undisturbed and a photographic record was taken.

5.3.2 Results and discussion

a. Elemental-S release from granules

There was a significant fertiliser source \times leaching time interaction on the release of elemental S from the granules. In all products there was a rapid release of S from the coat in the first 24 hours and a slower release thereafter. The release of elemental S from the PVA and LS products did not differ significantly and was greater than the release from the HF product (Figure 5.4). This was probably due to the differing response of each product to humidity as affected by the adhesive materials and coating process.

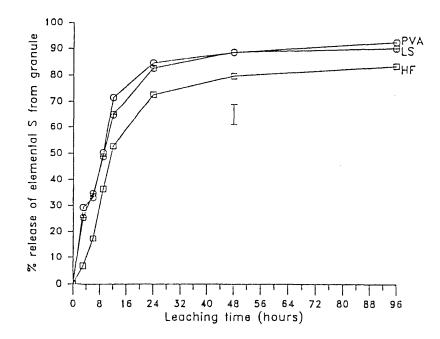
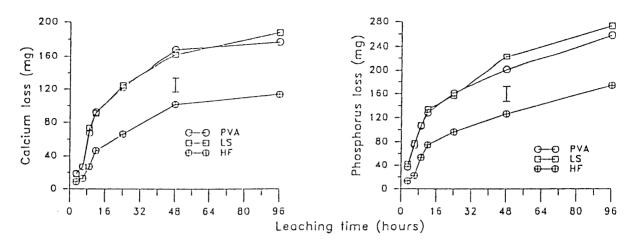
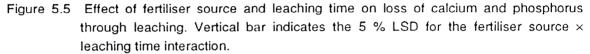


Figure 5.4 Effect of fertiliser source and leaching time on percentage of elemental S lost through leaching. Vertical bar indicates the 5 % LSD for the fertiliser source × leaching time interaction.

b. Phosphorus and calcium losses from granules

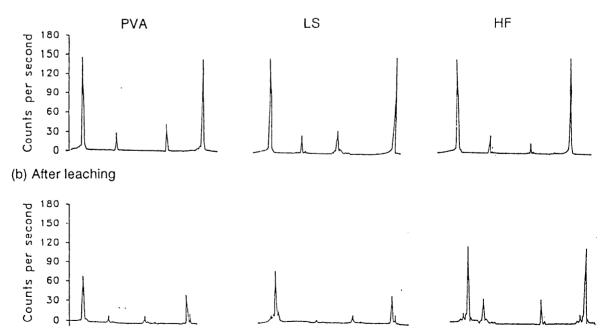
There was a significant fertiliser source × leaching time interaction in the loss of calcium and phosphorus from the coated fertilisers. The amount of calcium and phosphorus loss increased with time and the losses from the PVA and LS products did not differ significantly and were greater than the loss from the HF product (Figure 5.5). A greater loss of Ca and P from PVA and LS compared to HF was most likely due to the different dispersion rate of the granules under the leaching conditions. This was directly affected by the strength of the coat.



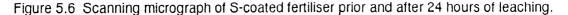


c. Microprobe scanning of granules

The microprobe scanning data from the coated fertilisers prior to and after 24 hours of leaching agreed with the chemical analysis of the granules. Prior to the leaching, no differences were observed in the percentage of S among PVA, LS and HF. The amount of S remaining in the granule, shown as counts per second, from HF was higher than from PVA and LS after 24 hours of leaching. This indicates that a higher amount of S was lost from the PVA and LS than from the HF granules (Figure 5.6).



(a) Before leaching

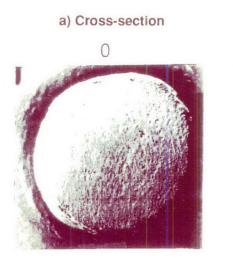


The results of these studies showed that the release rate of S from the HF fertiliser was lower than from the other sources. These differences are believed to be affected by the adhesive material used to bind the elemental S to the TSP. The greater release of S from the coat was followed by a greater rate of release of Ca and P from the TSP granules. These findings support the earlier studies (Chapter 3) where it was found that the oxidation of elemental S from the PVA and LS products was greater than that of HF. Release of S from fertilisers has been investigated by Janzen and Bettany (1986), who found differing rates of release from the different S sources, and suggested that this was related to the initial particle size of the product and the dispersion of the particles in the soil. The effect of temperature on the release of nutrients from coated fertiliser had been reported by Oertli and Lunt (1962). They found an approximate 100 % increase in initial release rate of nutrients when the temperature was increased from 10 to 20 °C. Brown *et al.* (1966) reported that a significant decrease in urea release was recorded with increasing coating thickness of resin from 9.0 to 13.2 %.

The photographs in Figure 5.7 show the cross-section of the granules (a) prior to and (b) after 24 hours in the leaching bed. After 24 hours, the elemental S remaining on the HF granule was greater than on the PVA and LS granules (Figure 5.7 b). The photographs show that in the cross-section (Figure 5.7 b) the HF granule was rougher than both the PVA and LS granules. It was observed that the moisture content of the HF granule was less than the other two when taken from the leaching bed, hence during the cutting process the sandpaper fractured the dry particle to a greater extent as compared to a relatively smooth surface for the initially moister PVA and LS granules.

The photographs in Figure 5.8 show the dispersion of the coated fertiliser in distilled water at different observation times. These photographs again illustrate the different properties of the coated fertilisers. This figure shows that HF was similar to SW and these sloughed less than the PVA and LS. This and earlier data suggest that PVA and LS are more effective coated fertilisers because of the faster granule dispersion.

CHAPTER 5 : PHYSICAL TESTS OF S-COATED FERTILISERS



TIME (HOURS)

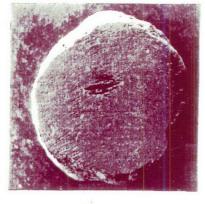




PVA

LS

HF



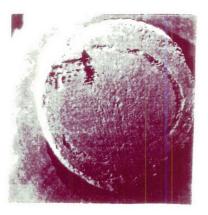
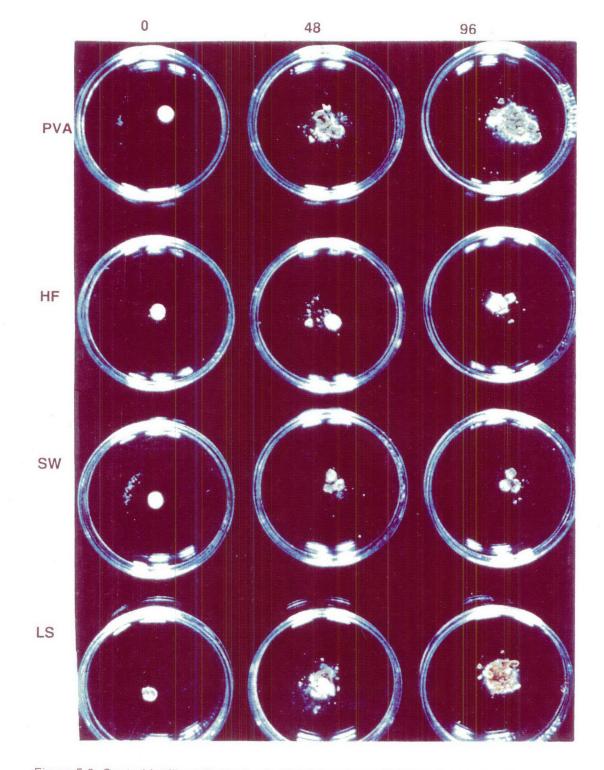




Figure 5.7 Electron micrographs of cross-section of coated fertilisers treated in a leaching bed for 24 hours.

CHAPTER 5 : PHYSICAL TESTS OF S-COATED FERTILISERS



TIME (HOURS)

Figure 5.8 Coated fertiliser dispersion in distilled water at different observation times.

CHAPTER 6

INTEGRATING DISCUSSION AND THE MATCHING OF

ELEMENTAL S RELEASE TO PLANT DEMAND

6.1 EFFECTIVENESS OF S TO CROPS AS AFFECTED BY AGRICULTURAL MANAGEMENT

To improve our understanding on the effectiveness of S for crops from different S sources under different soil conditions, the characteristic of S release from each source and the S requirement for each crop species needs to be known.

In this study, it was observed that there were differences between flooded and nonflooded rice in terms of growth rate but the S demand patterns were similar (Table 6.1). Although dry matter production was higher under flooded than under non-flooded conditions, the rate of S uptake by the crops was similar at each growth stage (Table 6.1) as was the rate of fertiliser S uptake (Table 6.1).

Growth stage	Water regime	growth rate (g pot ⁻¹ day ⁻¹)	S upt. rate (mg pot ⁻¹ day	
Frans AT	F	0.39	0.97	0.41
	NF	0.31	1.07	0.46
AT - MT	F	1.08	0.49	0.33
	NF	1.01	0.43	0.33
MT - M	F	0.63	0.48	0.32
	NF	0.43	0.39	0.29

Table 6.1 Dry matter yield, S content and rate of fertiliser S uptake by flooded and non-flooded rice fertilised with gypsum at 5 mg S kg¹ soil at AT, MT and M

Under field conditions, a higher amount of S could be lost through surface as well as downward movement of water. This could affect the efficiency of utilization of the applied fertiliser with the sulfate form being more susceptible to loss soon after application. This higher susceptibility to loss has to be balanced against the high initial S demand rate to produce tillers. The experiments reported here have shown that fertilisers such as sulfur bentonite (SB) and sulfur coated urea (SCU) do not release their S fast enough to meet the initial plant demand, although they have a higher residual value in the following crop.

The results of these experiments also indicate the importance of bonding strength when S is coated onto finished fertiliser products. The Hifert bonding process results in a coating which delays not only the S release rate (Tables 3.11, 3.27, 4.9) but also significantly reduces the rate of Ca and P movement from the TSP granules under laboratory leaching conditions (Figure 5.5).

Among the adhesive materials used in these studies, PVA and LS proved to be superior to SW. It is of interest to note that the initial rate of fertiliser S uptake from fertilisers where PVA and LS was used to bond the elemental S to the TSP was greater than from elemental S of the same particle size. This is most likely due to the enhanced oxidation rate of the elemental S when in intimate contact with P, as reported by Santoso (1989).

To maximize the effectiveness of fertiliser application many factors should be considered such as type and form of fertiliser used, crop species, method of application, soil conditions and other climatic factors.

In this study, it was observed that the sulfate form resulted in a higher recovery in crop 1 but a lower recovery in crop 2. To maximize the yield and efficiency of utilization of applied fertiliser in each crop, fertiliser application is required for each crop.

Although the slow release S fertilisers such as SB and SCU contributed more S for the second crop, the efficiency of fertiliser utilization was low because of the yield penalty in the first crop after application.

Similar S release patterns were also observed under pasture conditions. Under these conditions the sulfate form tends to contribute an excess amount of S in the early growth stages. This results in a higher S concentration in the pasture which can lead to increased losses in dung and urine. At the same time more S can be lost through leaching as measured in Chapter 4 (appendix 4.18). To improve fertiliser S efficiency in this system, controlled release S materials which are able to meet the S demand for the pasture throughout the year are more appropriate. In this study the TSP-S bonded with PVA or LS supplied S to the pasture at a faster rate than HF or SW up to 17 weeks after application and between 24 and 44 weeks HF was superior to these two products (Table 4.9). The performance of the LS and PVA could

possibly be improved by modifying the particle size of elemental S used in the coat. A mixture of finer and coarser particles could be used to meet this demand. It would be expected that the finer particles would provide S in the early stages and the larger particles would continue to supply S for a longer period.

Under field conditions the S oxidation will be significantly affected by climatic factors such as rainfall and temperature. Therefore, the effect of climatic factors from different areas on oxidation of elemental S with different particle sizes needs to be understood. Earlier studies at UNE by McCaskill and Blair (1988, 1989) developed a simulation model of S release from elemental S and this provided a means to test this hypothesis.

6.2 THE USE OF COMPUTER SIMULATION TO DEVELOP COATINGS TO MATCH S RELEASE RATE TO PLANT DEMAND

6.2.1 Introduction

Several authors have developed models to estimate the oxidation rate of elemental S (Shedley, 1982; McCaskill and Blair, 1989; Watkinson, 1989). In formulating his model Shedley (1982) used an empirical approach to formulate mathematical relationships, which was a multiplication of particle size, soil moisture, soil temperature, the nature of soil microbe populations and soil phosphate concentration functions. From this simulation, he estimated that 29 % of the elemental S (100 μ m particle size) was oxidized within a 1-year period. The low S release predicted by this model was suggested to be due to the lack of consideration of interactions among the environmental factors and to the fact that the simulation was drawn mainly from incubation studies.

McCaskill and Blair (1989) used a mechanistic approach to predict S release from elemental S. In this model, the S release was predicted based on the spherical particles and the equations used are presented in the literature review. This model was used in the present study to estimate the release of S from different particle sizes applied at different times of the year at four locations in Australia.

McCaskill and Blair (1989) predicted that with a constant release of 0.4 μ m day⁻¹ and under a non-restricted environment, particles less than 0.5 mm were almost completely oxidized after 12 months, but that 40 % of larger particles (> 1.0 mm) remained in the elemental form. From this prediction a mixture of fine and coarse particles was able to maintain the S status in the soil for a longer period to meet the S demand of crops and pastures.

In the present study, the model was used to estimate the S release and S demand for pastures as affected by different particle sizes of elemental S and different times of application. In each area, three different application times were simulated (Figures 6.1 and 6.2). This was based on fertiliser application times which are commonly used by farmers in each area.

The S demand for C₃ pastures estimated under non-limiting nutrients and different climatic conditions is presented in Figures 6.1 and 6.2. S release (kg ha day⁻¹) is expressed as the product of oxidation rate (% day⁻¹) and S-application rate (20 kg ha⁻¹). The release pattern is predicted from two particle sizes (100 and 200 μ m) and a mixture of 50% 100 μ m + 50% 200 μ m using a constant release rate of 0.3 μ m day⁻¹. This value was fitted in this model because of the overestimate of S oxidation compared to the oxidation rate which was observed in the earlier experiments (Chapter 3) when the attrition rate of 0.4 mm day⁻¹ was fitted in the model.

The model was run for four different temperature and moisture regimes around Australia : 2 tropical areas (Darwin and Cairns; North and North-east parts of Australia, respectively) and 2 temperate areas (Armidale, N.S.W. and Dalwallinu, Western Australia). The mean weekly climatic data used in this simulation were mean of maximum and minimum temperature, solar radiation, rainfall and evaporation, from 1969 to 1989. These data are presented in Figures 6.1 and 6.2.

6.2.2 S release from different particle sizes

The difference in oxidation rate of a 100 μ m diameter elemental S at different application times indicates the important effects of moisture and temperature on elemental S oxidation. In Armidale, for instance, the application of fertiliser in March resulted in a greater oxidation rate soon after application compared to the late application (in May). This was largely due to the higher temperatures in March and April than in May (Figure 6.1). The greater oxidation from the March application resulted in a lower oxidation rate during the spring period where the S demand of pastures is high. Fertiliser application in May is probably a better choice due to the higher S release during the spring period where the pastures require more S. The application of 200 μ m particles at this location was less suitable because of the slow S release during the Spring period.

By contrast, total oxidation of elemental S was less at Dalwallinu than at Armidale although the temperature at Dalwallinu is higher than that at Armidale. This was mainly due to moisture limitation over summer at Dalwallinu compared to that at Armidale.

In tropical areas (Darwin and Cairns), temperature was not a limiting factor to S oxidation. At both locations higher oxidation rates were predicted compared to the temperate areas particularly for coarser particles(Figure 6.2). The oxidation rate of 100 µm particles exceed the S demand early in the season, therefore application of larger particle sizes was more appropriate. The mixture of 50 % 100 μ m + 50 % 200 μ m particles was better able to match the S demand.

The effect of different particle sizes on oxidation of elemental S in different areas is presented in Table 6.2. The total predicted oxidation of elemental S with the 100 μ m particle size in the temperate area ranged between 86.3 % to 96.5 % in one calendar year (Table 6.2). Higher oxidation rates (99.9 to 100 %) were predicted in tropical areas.

From this simulation, S release from the 100 μ m particles was initially more rapid than from the 200 μ m or mixture treatments in all four environments. However, over longer periods, the amount of S released from finer particles would decrease as the amount of elemental S remaining falls rapidly. These simulations indicated that a finer particle size (100 μ m) of elemental S is more appropriate when this material is used to match S demand for pastures in temperate areas. In addition, a mixture of fine and coarse particles of elemental S (50 % 100 μ m + 50 % 200 μ m) is more suitable when this material is used in tropical areas.

 Table 6.2 Effect of different particle sizes on the annual oxidation of elemental S at four locations in Australia.

Location	Particle size (µm)	Total oxidation (%)
Armidale	100 Mixture 200	96.5 82.4 71.2
Dalwallinu	100 Mixture 200	86.3 69.2 56.7
Darwin	100 Mixture 200	99.9 91.6 83.6
Cairns	100 Mixture 200	100.0 94.5 88.4

The simulation undertaken in this study was not intended to supply all of S requirement for pastures as S mineralization from organic sources is an important source. In the earlier experiment (Table 4.5), the TSP + elemental S treatment and under non-leaching conditions derived 27.8 % of S in the pasture tops from the fertiliser at the first harvest and this increased up to 50.5 % in the second harvest and then declined in the following harvests.

The simulation showed that the model can be used to predict the oxidation rate of elemental S in order to match S supply from fertilisers to the S demand of pastures. There are several disadvantages associated with the handling and application of fine particle of elemental S. These problems can be solved by combining elemental S with TSP granules through the

coating process which has been developed in this study. In the coating process a mixture of fine and coarse particle can be used to match the S demand for crops or pastures in a wide range of different climatic conditions. In the earlier experiment (Chapter 3) it was found that S-coated TSP (with PVA or LS as a binding agent) was as effective as gypsum in increasing grain yield of rice.

In the pasture experiment (Chapter 4) and under non-leaching conditions it was observed that there was no significant difference in total pasture yield (Figure 4.3) or S content (Figure 4.4) recorded between the elemental S and gypsum treatments over one year period. In contrast, under leaching conditions, a significantly higher total yield (Figure 4.3) and S content (Figure 4.4) of pastures was recorded with elemental S than with gypsum. This difference was most likely due to the higher S loss through leaching in the gypsum treatment.

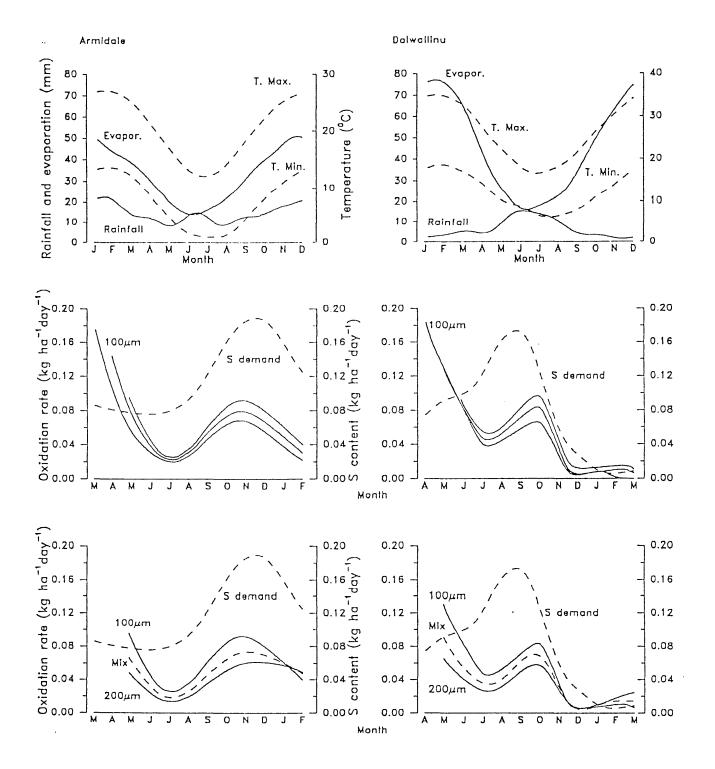


Figure 6.1 Prediction of S demand of pastures under non-limiting nutrient conditions and oxidation of elemental S as affected by temperature, moisture, time of application and particle sizes in two temperate locations in Australia.

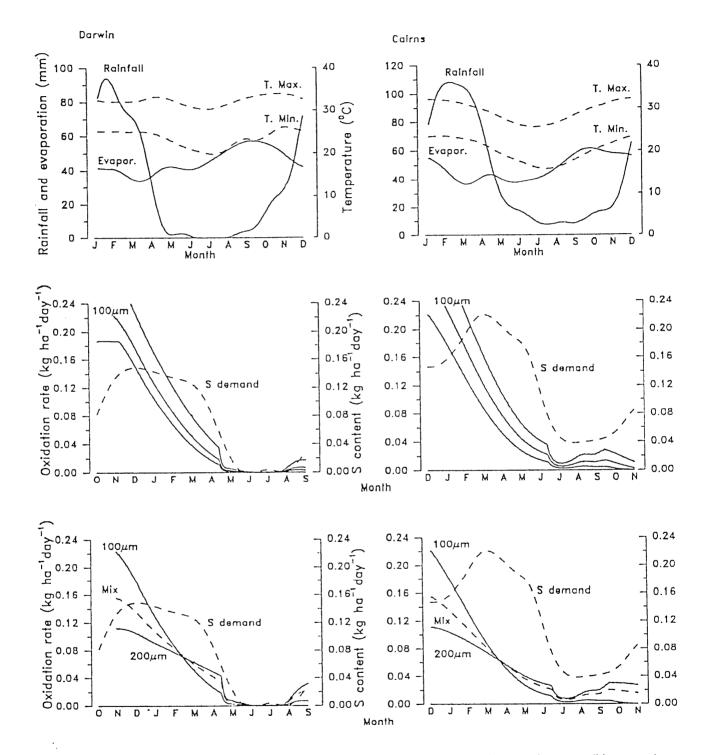


Figure 6.2 Prediction of S demand of pastures under non-limiting nutrient conditions and oxidation of elemental S as affected by temperature, moisture, time of application and particle sizes in two tropical locations in Australia.

These series of experiments have identified two S coated TSP candidate products where litter polyvinyl alcohol (PVA) or calcium ligno sulfonate (LS) is used as an adhesive to bind the elemental S to the fertiliser. Patents have been obtained for this invention and studies are in progress to scale up the process for a commercial venture. The combination of glasshouse trials, laboratory evaluation, microscopic and microprobe scans and computer simulation presented in this study have developed a fertiliser product that firstly matches plant requirements and secondly is mechanically robust.

This study complements earlier work (Samosir, 1981; Santoso, 1989; Chaitep, 1990) and studies still in progress at UNE to match fertiliser release rates to plant demand. This will lead to more efficient use of nutrients, better economies of fertiliser use and less environmental problems from nutrient leaching and runoff.

This approach should be extended to nitrogenous (urea) and NP fertilisers (DAP, MAP) where S coating may serve not only to supply S to the plant but modify the release pattern of the other nutrients.

6.3 FUTURE RESEARCH AND DEVELOPMENT REQUIRED

To confirm the results from the glasshouse experiment, further field trials are required. These trials should involve S rate studies using sulfate forms such as gypsum or ammonium sulfate and elemental S coated materials. For flooded rice it would seem to be most appropriate to concentrate on P-S products as Lefroy (1989) argues that these are more appropriate than N-S sources. These studies would provide S response curves for each product under the prevailing conditions to enable a comparison of the effectiveness of the different sources. This would allow a horizontal comparison and substitution value to be calculated. These studies should be undertaken at well defined field sites where environmental conditions can be adequately monitored. This would include soil temperature, moisture and water movement (both in floodwater and through the profile). In these studies S would need to be applied to each crop and the residual value of fertiliser calculated by the rate of movement of the response curves to the left from year to year.

The S requirement for different species of crops needs to be considered. This becomes particularly complex in situations where flooded rice is followed by non-flooded crops such as grain legumes or corn.

In pastures, field evaluations become even more complex because of the need to graze the plot to obtain realistic nutrient cycling conditions. This means that it would be unrealistic to examine a series of products applied over a range of rates. For this reason a rate which is towards the upper end of the response curve needs to be chosen for the evaluation.

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APPENDICES

3.1 Examples of variance analysis using NEVA computer program.

(a) The effect of S application rate on grain yield under flooded conditions.

FILE GGRI.OUT CREATED BY NEVA VERSION 3.3 ON 9/3/88 AT 13H 34M 1 NEVA EXF (THE EFFECT OF 5 RATE ON GRAIN YIELD) 2 FACTORS R3 T4 2 FACTORS 12 ITEMS R-REPLICATE T=S RATE (0, 5, 10, 20) * * • • 12 ITEMS
 C R-REPLICATE T-S RAT
 DATA
 16.485 16.003 14.226
 25.151 26.930 24.400
 30.683 30.777 30.678
 8 35.615 34.569 36.529
 BND **** # 12 ITEMS, 0 MISSING OBS 12 ITEMS, MIN MAX 14.226000 MBAN 36.529000 B.D. 26.837170 SUM 7.793984 322,046000 EFFECTS R T RT/ Aov means d5 res plot 10 ÷. 11 SOURCE DF SUM OF SQUARES MBAN SQUARE F PROB GRAND MEAN 8642.80400000 0.87500000 8642.80400000 7140.4820 0.00000*** 0.3615 0.71086 1 0.3615 0.71086 181.7779 0.00000*** 220.02300000 1.21039500 660.06900000 7.26237000 3 6 RT/ RAW SS 9311.01000000 668.20610000 COR SS DFa 6 SD OF MEANS BASED ON PLOT BRROR 1.100180 GRAND MEAN MEANS OF 12, SD= 0.31759 5PC LSD= 1.09951 1PC LSD= 1.66541 26.83717 MEANS OF 4. SD= 0.55009 5PC LSD= 1.90441 R 1PC LSD= 2.88457 R 1 R 2 R 3 26.98350 27.06975 26.45825 MEANS OF 3. SD= 0.63519 т 5PC LSD= 2.19902 1PC LSD= 3.33081 T 1 T 2 T 3 T 4 15.57133 25.49367 30.71267 35.57100 2 3 5PC DUNCANS MULTIPLE RANGE 2.19808 2.27827 4 2.31826 HAX. ABS. RESIDUAL 1.3369 0.7673 0.1991 -0.9664 -0.4890 1.2038 -0.7147 -0.1760 -0.1682 0.3442 -0.1023 -1.2346 1.3369 PREQUENCY PLOT OF ABSOLUTE RESIDUALS AGAINST CLASS MRANS OF X. RMS DENOTES THE ROOT MEAN SQUARE RESIDUAL IN EACH CLASS. FREQUENCY MEAN X RMS RESIDUAL 0.0000 0.6685 1.3369 3 15.5713 0.7217 . 1 1 . RMS 0 1 . RMS 2 24.7755 0.6124 . 1 . . 1 26.9300 1.2038 . . RMS . з 30.7127 0.2434 . 2 1 RMS . . 1 1 35.5710 з 1.0523 . 1 RMS ---------SLOPE OF REGRESSION OF LOG ABSOLUTE RESIDUAL ON LOG(X) IS -0.3982 0.8217 WITH DF = 11. APPROXIMATE S.D. OF SLOPE IS 12 STOP # END OF NEVA JOB 12 LINES INPUTTED 76 LINES OUTPUTTED

APPENDIX

(b) The effect of different S sources on relative grain yield under flooded conditions.

```
FILE GRCR.OUT CREATED BY NEVA VERSION 3.3
ON 9/ 3/88 AT 12H 34M
1 NEVA EXF ( RELATIVE GRAIN YIELD UNDER FLOODED CONDITIONS )
2 FACTORS R3 T10
 #
                  2 FACTORS
30 ITEMS
      ..
          30 ITRNB
DATA
53.272 51.997 46.372
100.000 100.000 100.000
88.459 79.231 93.856
76.698 87.845 87.665
74.970 71.284 62.028
82.254 94.593 81.443
95.030 98.778 89.703
100.766 79.797 76.977
93.579 92.527 95.192
59.378 72.574 52.549
END
0 MISSING (
      * *
        3
        4
5
 *****
      9
10
       11
12
       13
14
           30 ITEMS, 0 MISSING OBS
         30 ITEMS, MIN 46.372000 MHAN
NAX 100.766000 S.D.
                                                                     81.360570 SUM
                                                                                                  2440.817000
                                                                81.360570
16.319570
 # 15 BFFRCTS R T RT/
# 16 AOV MEANS D5 RBS PLOT
 SOURCE
                           DF
                                                SUM OF SQUARES
                                                                                      MBAN SOUARB
                                                                                                                   P
                                                                                                                                  PROB
                                 1
2
9
18
                                                                                                          4144.1680 0.00000***
1.2140 0.32019
15.6388 0.00000***
                                                                                   198586.200000
 GRAND MEAN
                                                 198586.200000
                                                      116.350000
6744.633000
862.550000
                                                                                      58.175000
749.403700
47.919450
 RT/
                                                 206309.800000
7723.531000
 RAN SS
COR SS
                                                         6.922387
                                                                          DF = 18
 SD OF MEANS BASED ON PLOT BEROR
                       MBANS OF 30. 8D- 1.26385
                                                                             5PC LSD- 3.75809
                                                                                                                     1PC LSD= 5.14510
 GRAND MRAN
                  81.36057
                        MEANS OF 10. SD- 2.18905
                                                                              5PC L8D= 6.50921
                                                                                                                       1PC LSD= 8,91158
 R
                  R1 R2 R3
82.64059 82.86259 78.57850
                        MBANS OF 3. 8D= 3.9966
                                                                              5PC LSD= 11.8841
                                                                                                                       1PC LSD= 16.2702
т
        T1 T2 T3 T4 T5 T6 T7 T8 T9 T10
50,5470 100,0000 87,1820 84,7360 69,4273 86,0967 94,5037 85,8467 93,7660 61,5003
  SORTED MEANS
50.5470 61.5003 69.4273
                                                        84.7360
                                                                           85.8467
                                                                                            86.0967
                                                                                                               87.1820
                                                                                                                                 93.7660
                                                                                                                                                    94.5037
                                                                                                                                                                     100.0000
                                                                                            7
                                         4
                                                                               6
                                                                                                                   8
      2
                        з
                                                      5
                                                                                                                                     9
                                                                                                                                                      10
 5PC DUNCANS MULTIPLE RANGE
11.8747 12.4593 12.8296
                                                    13.0852
                                                                           13.2717
                                                                                        13.4125
                                                                                                          13.5213
                                                                                                                             13.6066
                                                                                                                                                    13.6741

        MAX.
        ABS.
        REBIDUAL
        13.639

        1.445
        -0.052
        -1.393

        -1.280
        -1.502
        2.782

        -0.003
        -9.453
        9.456

        -7.318
        1.607
        5.711

        4.263
        0.355
        -4.617

        -5.123
        6.994
        -1.872

        -0.754
        2.772
        -2.019

        13.639
        -7.552
        -6.088

               -2.741 9.572
    -1.467
                         4.208
FREQUENCY PLOT OF ABSOLUTE RESIDUALS AGAINST CLASS MEANS OF X.
RMS DENOTES THE ROOT MEAN SQUARE RESIDUAL IN EACH CLASS.
              MEAN X RMS RESIDUAL 0.0000
                                                                                               13.6393
FREQUENCY
                                                                              6.8197
                                                                    _____
      4
                   51.0475
                                            3.2439
                                                           1 2
                                                                                1.
                                                                                                         .
                                                                     вма
      2
                   60.7030
                                            4.0556
                                                                      1
                                                                       1 1
RMS
                                                                                                          .
      2
                   71.9290
                                            6.7728
                                                            .1
                                                                                            1
                                                                                                         .
                                                                                 RM 9
      7
                   79.0529
                                            6.3798
                                                                      1 1 1 .11
                                                           . 1
                                                                                          1
                                                                                                          .
                                                                               RMR
      4
                   88.4180
                                           3.1334
                                                          1 2
                                                                               1.
                                                                   ิหหร
                                                          . 13 3 1 1 1
RMS
    11
                   96.7564
                                            5.8124
                                                                                                        1
RMS
 SLOPE OF REGRESSION OF LOG ABSOLUTE RESIDUAL ON LOG(X) IS
APPROXIMATE S.D. OF SLOPE IS
                                                                                                 0.9903
1.2258 WITH DF = 29.
# 17 STOP
END OF NEVA JOB
17 LINES INPUTTED
63 LINES OUTPUTTED
```

Yield component	growth		Sı	rate (kg ha ⁻¹)	
	stages	0	5	10	20
Number of tillers	31 dat 45 dat AT MT M	1.3 b ^A 2.0 c 3.0 c 7.0 c 9.0 c	2.7 b 5.7 b 8.3 b 10.7 b 12.7 b	4.7 a 8.0 a 12.0 b 15.3 a 16.0 ab	5.0 a 8.7 a 13.3 a 18.0 a 18.0 a
Number of panicles	М	8.7 c	12.0 b	15.0 a	17.7 a
Number of grain per panicle	М	80.4 b	105.4 a	99.0 a	98.1 a
Dry weight of grain per panicle (g)	Μ	1.6 b	2.1 a	2.0 a	2.0 a
Dry weight of 100 grains (g)	М	2.0 a	2.0 a	2.1 a	2.1 a
Dry weight of empty grains (g pot ⁻¹)	Μ	1.1 a	1.4 a	1.3 a	1.3 a
Percentage of empty grain (%)	М	6.5 a	4.2 b	4.9 b	4.8 b
Harvest index	М	0.36 a	0.37 a	0.34 a	0.35 a

3.2 The effect of S-application rate on yield components at different growth stages under flooded conditions.

^A Data followed by the same letter in the same row are not significantly different (P < 0.05) (Duncan's multiple range test).

3.3 The effect of S-application rate on extractable S (ES) (mg pot⁻¹) and organic S (OS) (mg pot⁻¹) at different growth stages under flooded conditions.

	AT		M	1T	N	Л
Treatment	ES	OS	ES	OS	ES	OS
	(mg p	ot ⁻¹)	(mg	pot ⁻¹)	(mg po	ot -1)
С	38.5 d ^A	630.4 c	20.3 d	602.1 b	14.5 c	575.7 a
G1	49.9 c	638.9 bc	25.0 c	608.7 b	15.8 bc	579.2 a
G ₂	61.8 b	643.3 ab	32.3 b	616.8 ab	17.9 ab	582.1 a
G ₃	70.7 a	652.8 a	37.0 a	627.8 a	19.6 a	585.0 a

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

Source of S	No. of panicles	Dwt. of grains panicle ⁻¹ (g)	Dwt. of 100 grain (g)	percentage of empty grain (%)	Harvest index
G	15.0 a ^A	2.1 a	2.1 a	4.2 b	0.34 a
PVA	14.0 ab	2.1 a	2.1 a	5.0 b	0.35 a
LS	13.3 bc	2.2 a	2.0 a	4.6 b	0.36 a
HF	11.0 d	2.4 a	2.0 a	4.5 b	0.39 a
SW	11.7 d	2.3 a	2.1 a	4.5 b	0.38 a
US	12.0 cd	2.2 a	2.0 a	4.8 b	0.36 a
SCU	8.0 e	2.7 a	2.0 a	5.0 b	0.38 a
E	12.3 cd	2.2 a	2.0 a	4.9 b	0.36 a
SB	8.3 e	2.3 a	2.1 a	5.4 ab	0.38 a
C	8.0 e	2.0 a	1.1 a	6.5 a	0.36 a

3.4 The effect of S sources on yield components at maturity under flooded conditions.

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.5 The effect of S sources on relative yield of plant components at different growth stages under flooded conditions.

(a) AT

Source	Relati	ive yield (%)	total
of S	stem + leaf	root	
G	100.0 a ^A	100.0 a	100.0 a
PVA	76.8 b	65.5 b	72.6 b
LS	72.4 b	55.4 c	66.1 c
HF	58.5 c	33.9 e	49.2 e
SW	56.9 c	37.3 e	49.5 e
US	60.4 c	34.0 e	50.5 de
SCU	32.1 d	13.1 g	25.0 f
E	63.2 c	44.8 d	56.3 d
SB	35.3 d	23.3 f	30.8 f
С	28.9 d	17.8 fg	24.8 f

A Data followed by the same letter in the same column are not significantly different

(P < 0.05) (Duncan's multiple range test).

(b)	M

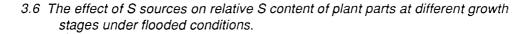
Source	Rela	tive yield (%)	total
of S	stem + leaf	root	
G	100.0 a ^A	100.0 a	100.0 a
PVA	94.4 ab	87.9 ab	92.2 ab
LS	90.6 a-c	86.5 ab	89.0 a-c
HF	64.4 de	64.0 cd	64.4 d
SW	78.6 cd	69.3 bc	75.6 cd
US	78.8 cd	70.6 bc	74.8 cd
SCU	56.1 ef	37.2 e	49.7 e
E	83.3 bc	76.8 bc	80.9 bc
SB	51.3 ef	44.6 de	49.0 e
С	45.3 f	35.1 e	41.8 e

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(c) M

Source of S	grain	Relative yield (stem + leaf	root	total
G	100.0 a ^A	100.0 a	100.0 a	100.0 a
PVA	94.5 ab	94.4 a	78.4 bc	90.3 ab
LS	93.8 ab	91.6 ab	81.0 b	89.7 ab
HF	86.1 b	75.7 c	61.8 cd	75.7 c
SW	85.8 b	78.7 c	65.4 bc	77.9 c
US	84.7 b	82.0 bc	73.5 bc	80.8 c
SCU	69.4 c	64.3 d	46.7 de	61.7 d
E	87.2 b	84.2 bc	74.4 bc	82.8 bc
SB	61.5 cd	55.1 de	47.3 de	55.3 de
С	50.7 d	50.9 e	39.5 e	47.9 e

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).



(a) AT

Source	Relative	S content (%)	total
of S	stem + leaf	root	
G	100.0 a ^A	100.0 a	100.0 a
PVA	86.4 b	75.8 b	83.8 b
LS	78.5 bc	56.7 c	72.4 c
HF	65.0 d	38.7 e	57.9 d
SW	63.0 d	43.0 de	57.3 d
US	66.8 d	42.7 de	60.0 d
SCU	35.9 e	18.0 f	31.2 e
E	70.9 cd	49.8 cd	64.9 cd
SB	39.0 e	26.0 f	35.5 e
С	32.5 e	23.8 f	30.1 e

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(b) MT

Source	Relative	S content (%)	total
of S	stem + leaf	root	
G	100.0 a ^A	100.0 a	100.0 a
PVA	92.7 ab	85.9 ab	90.9 ab
LS	88.2 b	73.9 bc	84.4 b
HF	68.1 c	51.1 d	63.3 d
SW	70.8 c	60.7 cd	67.8 cd
US	73.6 c	61.7 cd	70.4 cd
SCU	52.0 d	35.2 e	47.5 e
E	77.0 c	60.1 cd	72.5 c
SB	48.1 de	36.0 e	45.0 e
С	42.5 e	30.9 e	39.5 e

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(c) M

Source of S	grain	Relative S con stem + leaf	tent (%) root	total
G	100.0 a ^A	100.0 a	100.0 a	100.0 a
PVA	94.2 ab	94.2 a	94.8 a	95.2 ab
LS	89.9 b	90.5 a	92.0 a	91.9 b
HF	76.6 c	70.0 b	70.4 b	73.9 c
SW	74.4 c	76.1 b	71.2 b	75.7 c
US	79.5 c	74.4 b	72.8 b	77.4 c
SCU	62.7 d	56.0 c	57.4 c	60.0 d
E	79.4 c	79.0 b	73.0 b	79.3 c
SB	54.9 d	53.5 cd	58.0 c	55.7 d
С	45.0 e	43.5 d	38.9 d	42.0 e

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.7 The effect of different S sources on extractable S (ES) at different growth stages under flooded conditions.

Source	AT Extra	actable S (mg p	ot ⁻¹)
of S		MT	M
G	61.8 a ^A	32.3 h	17.9 j
PVA	54.8 b	33.1 h	19.2 j
LS	53.4 b	31.7 h	18.8 j
HF	45.1 de	30.4 h	19.5 j
SW	48.1 cd	30.4 h	21.2 j
US	46.4 de	31.9 h	18.2 j
SCU	41.9 f	25.5 i	24.5 i
E	49.8 c	32.0 h	18.7 j
SB	43.5 ef	25.8 i	26.4 j
С	38.5 g	20.3 j	14.5 k

^A Data followed by the same letter are not significantly different (P < 0.05) (Duncan's multiple range test).

APPENDIX

Source	Orga	anic S (mg pot ⁻¹)	М
of S	AT	MT	
G	643.3 a ^A	616.8 bc	582.1 e
PVA	637.6 a	613.6 c	583.0 e
LS	636.4 a	613.9 c	584.0 e
HF	634.3 a	612.0 c	584.3 e
SW	633.7 a	615.3 c	585.5 e
US	634.2 a	613.1 c	583.8 e
SCU	635.0 a	616.0 c	589.0 de
E	635.9 a	615.1 c	584.8 e
SB	634.7 a	612.4 c	588.1 de
С	630.4 ab	602.1 cd	575.7 e

3.8 The effect of S sources on organic S (OS) at different growth stages under flooded conditions.

^A Data followed by the same letter are not significantly different (P < 0.05) (Duncan's multiple range test).

3.9 The residual effect of S-application rate on yield components under flooded conditions.

Yield component	S rate (kg ha ⁻¹)			
	0	5	10	20
Number of tillers	8.0 A	8.7 ab	9.0 ab	10.7 a
Number of panicles	7.3 b	8.3 ab	8.7 ab	10.0 a
Number of grains	52.3 a	60.0 a	61.4 a	60.6 a
per panicle				
Dry weight of	1.06 a	1.20 a	1.24 a	1.22 a
grain panicle ⁻¹ (g)				
Dry weight of	2.02 a	2.00 a	2.02 a	2.02 a
100 grains (g)				
Percentage of	7.3 a	5.8 ab	5.7 ab	5.5 b
empty grain (%)				
Harvest index	23.5 b	27.9 ab	28.2 ab	29.1 a

A Data followed by the same letter in the same row are not significantly different (P < 0.05) (Duncan's multiple range test).

3.10 The residual effect of S-application rates on extractable S (ES) and organic S (OS) under flooded conditions.

Treatment	ES (mg pot ⁻¹)	OS (mg pot ⁻¹)
С	6.9 b ^A	404.6 a
G ₁	7.7 ab	413.9 a
G ₂	8.4 ab	416.1 a
G ₃	10.5 a	421.5 a

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).</p> 3.11 The residual effect of S-application rate on the percentage of fertiliser S incorporated into the extractable S (ES) and organic S (OS) fractions under flooded conditions.

S rate (kg ha ⁻¹)	Recovery of fer ES	tiliser S (%) OS
0		
5	0.48 a ^A	24.9 a
10	0.91 a	31.9 a
20	1.74 a	45.1 a

^A Data followed by the same letter in the same column are not significantly different (*P* < 0.05) (Duncan's multiple range test).

Source of S	e No. of tillers	No. of panicles	Dwt. of grains panicle ⁻¹ (g)	Dwt. of 100 grain (g)	percentage of empty grain (%)	Harvest index
G	9.0 bc ^A	8.7 bc	1.24 a	2.02 a	5.7 ab	0.28 ab
PVA	9.7 a-c	9.3 a-c	1.18 a	2.02 a	5.9 ab	0.28 ab
LS	10.3 a-c	9.7 a-c	1.16 a	2.03 a	5.2 b	0.27 ab
HF	10.7 a-c	10.3 ab	1.14 a	2.03 a	4.8 b	0.28 ab
SW	11.3 ab	11.0 ab	1.08 a	2.03 a	5.6 ab	0.29 a
US	10.3 a-c	10.0 ab	1.15 a	2.01 a	6.0 ab	0.28 ab
SCU	12.0 a	11.3 a	1.13 a	2.02 a	4.9 b	0.28 ab
E	10.0 a-c	9.7 a-c	1.16 a	2.03 a	6.2 ab	0.28 ab
SB	12.3 a	11.7 a	1.17 a	2.02 a	4.8 b	0.29 a
С	8.0 c	7.3 c	1.06 a	2.02 a	7.3 a	0.23 b

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.13 The residual effect of different sources of S on extractable S (ES), organic S (OS) and acetone-extractable elemental S (AES) under flooded conditions.

Source	ES	OS	AES
of S	(mg pot ⁻¹)	(mg pot ⁻¹)	(mg pot ⁻¹)
С	6.9 c ^A	404.6 b	
G	8.4 bc	416.1 ab	
PVA	8.5 bc	418.7 ab	0.6 a
LS	8.7 bc	419.7 ab	0.7 a
HF	10.3 a-c	423.7 ab	0.9 a
SW	9.7 a-c	425.8 ab	1.1 a
US	9.4 a-c	421.1 ab	1.0 a
SCU	11.9 ab	427.3 a	1.2 a
E	9.3 a-c	419.1 ab	0.7 a
SB	12.8 a	429.1 a	1.8 a

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test). 1

Yield component	Growth		S r	ate (kg ha ⁻¹)	<u></u>
•	stages	0	5	`10 ´	20
Number of tillers	31 dat 45 dat AT MT	2.3 c ^A 5.0 c 7.7 c 10.3 b	3.3 bc 6.7 bc 9.7 bc 12.7 b	5.0 ab 9.0 ab 13.3 ab 17.7 a	5.7 a 9.7 a 15.0 a 19.0 a
Number of panicles Number of grain per panicle	M M M	11.6 b 10.7 b 54.0 a	13.0 b 12.3 b 56.7 a	18.3 a 17.7 a 44.6 a	19.7 a 19.0 a 47.4 a
Dry weight of grains per panicle (g)	М	1.1 a	1.1 a	0.9 a	1.0 a
Dry weight of 100 grains (g)	М	2.0 a	2.0 a	2.1 a	2.1 a
Percentage of empty grains (%)	М	6.2 a	5.0 b	4.5 b	3.6 c
Harvest index	М	0.22 a	0.23 a	0.22 a	0.23 a

3.14 The effect of S-application rate on yield components at different growth stages under non-flooded conditions.

A Data followed by the same letter in the same row are not significantly different (P < 0.05)

(Duncan's multiple range test).

3.15 The effects of S-application rate on extractable S (ES) and organic S (OS) at different growth stages under non-flooded conditions.

Treatment	A ES (mg po	OS	ES	MT OS pot ⁻¹)	M ES (mg po	OS
C	35.2 d ^A	628.8 c	19.6 c	608.1 b	11.1 c	585.1 a
G ₁	42.9 c	639.3 bc	23.6 c	616.8 ab	12.7 bc	602.0 a
G ₂	58.7 b	648.9 ab	30.2 b	622.5 ab	14.1 b	611.5 a
G ₃	69.3 a	663.6 a	34.2 a	630.8 a	17.7 a	619.4 a

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.16 The effect of S sources on yield components at maturity under non-flooded conditions.

Source of S	No. of panicles	Dwt. of grains panicle ⁻¹ (g)	Dwt. of 100 grains (g)	Percentage of empty grains (%)	Harvest index
G	17.7 a ^A	0.9 a	2.04 a	4.5 b	0.22 a
PVA LS HF SW	16.0 ab 15.7 a-c 12.3 d-f 13.0 c-f	0.9 a 1.0 a 1.1 a 1.1 a	2.01 a 2.03 a 2.02 a 2.03 a	5.2 ab 5.1 ab 5.4 ab 5.2 ab	0.22 a 0.22 a 0.23 a 0.22 a
US SCU	13.7 b-e 15.3 a-c	1.1 a 1.0 a	2.02 a 2.02 a	5.6 ab 5.1 ab	0.22 a 0.21 a
E SB	15.0 a-d 11.7 ef	1.0 a 1.1 a	2.01 a 2.03 a	5.2 ab 5.6 ab	0.22 a 0.22 a
С	10.7 f	1.1 a	2.01 a	6.2 a	0.21 a

A Data followed by the same letter in the same row are not significantly different (P < 0.05)

(Duncan's multiple range test).

3.17 The effect of S source on relative yield of plant components at different growth stages under non-flooded conditions.

(a) AT

Source	Belati	ve yield (%)	
of S	stem + leaf	root	total
G	100.0 a ^A	100.0 a	100.0 a
PVA LS HF SW	83.9 b 82.6 bc 76.2 b-d 80.3 b-d	91.6 ab 76.8 bc 67.0 cd 67.7 cd	84.0 b 79.7 b 71.0 bc 74.2 bc
US SCU	76.1 b-d 76.9 b-d	62.6 cd 73.1 b-d	71.3 bc 75.8 bc
E SB	80.7 b-d 68.6 cd	67.4 cd 52.7 d	74.4 bc 63.0 c
С	67.0 d	62.5 cd	63.4 c

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(b) MT

Source		tive yield (%)	
of S	stem + leaf	root	total
G	100.0 a ^A	100.0 a	100.0 a
PVA LS HF SW	93.3 ab 87.5 a-c 79.4 bc 83.3 bc	90.0 ab 88.1 ab 69.4 cd 73.1 b-d	92.8 ab 87.7 bc 77.3 c-e 81.2 b-d
US SCU	82.9 bc 87.6 a-c	76.3 b-d 81.1 bc	81.6 b-d 86.2 bc
E SB	90.6 ab 73.8 cd	84.5 a-c 68.2 cd	89.4 ab 72.7 de
С	64.1 d	61.9 d	63.5 e

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(c) M

Source of S	grain	Relative yield (stem + leaf	(%) root	total
G	100.0 a ^A	100.0 a	100.0 a	100.0 a
PVA	95.0 ab	95.1 a	94.5 a-c	94.9 ab
LS	94.2 a-c	93.8 ab	96.3 ab	94.1 ab
HF	85.9 cd	82.5 cd	83.3 cd	83.3 de
SW	86.3 c	84.2 cd	86.0 bcd	84.8 d
US	88.0 bc	87.0 bc	88.5 a-c	87.3 cd
SCU	93.2 a-c	93.7 ab	93.2 a-c	93.5 a-c
E	93.0 a-c	93.0 ab	92.9 a-c	93.0 a-c
SB	77.8 de	77.0 de	76.3 de	77.0 ef
С	71.6 e	73.6 e	69.7 e	72.8 f

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.18 The effect of S sources on relative S content of plant components at different growth stages under non-flooded conditions.

(a) AT

Source	Relative	S content (%)	total
of S	stem + leaf	root	
G	100.0 a ^A	100.0 a	100.0 a
PVA	84.9 b	74.5 b	82.1 b
LS	81.2 bc	74.2 b	79.8 bc
HF	72.5 cd	62.6 c	69.6 de
SW	73.3 cd	63.3 c	70.5 de
US	76.3 b-d	65.7 bc	73.4 c-e
SCU	75.0 b-d	64.4 bc	72.1 de
E	78.5 b-d	70.3 bc	76.3 b-d
SB	68.3 de	61.3 c	66.0 ef
С	60.9 e	64.4 bc	61.5 f

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(b) MT

Source of S	Relative stem + leaf	S content (%) root	total	
G	100.0 a ^A	100.0 a	100.0 a	_
PVA	94.0 ab	83.1 b	91.7 ab	
LS	90.7 bc	76.2 bc	87.5 bc	
HF	81.2 de	67.5 c	78.3 cd	
SW	76.0 ef	68.8 c	74.5 de	
US	83.5 c-e	70.9 c	80.8 cd	
SCU	89.3 b-d	68.7 c	85.0 bc	
E	88.9 b-d	73.7 bc	85.6 bc	
SB	68.0 fg	65.8 c	67.5 ef	
С	62.0 g	65.0 c	62.5 f	

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

(c) M

Source of S	grain	Relative S cont stem + leaf	ent (%) root	total
G	100.0 a ^A	100.0 a	100.0 a	100.0 a
PVA	95.6 ab	96.3 ab	92.1 ab	95.5 ab
LS	93.1 a-c	95.1 ab	89.2 bc	93.6 a-c
HF	82.9 c-e	82.8 cd	73.9 e	81.4 de
SW	84.5 b-d	87.6 b-d	77.4 de	85.0 cd
US	`81.5 de	88.9 bc	76.2 de	84.8 c-e
SCU	93.4 a-c	93.6 ab	86.8 bc	92.4 a-c
E	90.3 a-d	90.6 a-c	83.5 cd	89.3 b-d
SB	71.9 ef	79.0 de	70.0 ef	75.6 ef
С	62.9 f	70.6 e	64.8 f	67.5 f

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

Source	Extractable	sulphate S (mg	pot ⁻¹)
of S	AT	MT	M
G	58.7 a ^A	30.2 fg	14.1
PVA	56.7 ab	34.2 ef	14.8 kl
LS	55.2 a-c	33.0 e-g	15.3 j-l
HF	47.4 d	31.4 e-h	17.5 i-k
SW	49.5 cd	32.8 e-g	18.0 ij
US	48.7 cd	31.1 fg	17.3 i-k
SCU	50.5 b-d	32.2 e-g	15.5 j-l
E	52.1 a-d	33.5 e-g	16.2 j-l
SB	46.5 d	29.1 g	22.3 h
С	35.2 e	19.6 hi	11.1 m

3.19 The effect of different S sources on extractable S (ES) at different growth stages under non-flooded conditions.

^A Data followed by the same letter are not significantly different (P < 0.05) (Duncan's multiple range test).

3.20 The effect of S sources on organic S (OS) at different growth stages under non-flooded conditions.

Source	Orga	nic S (mg pot ⁻¹)	М
of S	AT	MT	
G	648.9 a ^A	622.5 a-f	611.5 b-g
PVA	643.1 ab	620.2 a-f	603.7 e-g
LS	638.5 a-c	621.5 a-f	606.8 c-g
HF	632.4 a-e	628.4 a-f	598.1 fg
SW	634.4 a-e	625.9 a-f	604.3 a-g
US	635.3 a-e	624.9 a-f	602.9 e-g
SCU	638.4 a-c	622.2 a-f	596.7 fg
E	637.7 a-d	625.5 a-f	602.1 e-g
SB	629.9 a-f	624.1 a-f	615.9 a-g
С	626.8 b-f	608.1 c-g	585.1 g

A Data followed by the same letter are not significantly different

(P < 0.05) (Duncan's multiple range test).

3.21 The residual effect of S-application rate on yield components under non-flooded conditions.

Yield component		S rate	(kg ha-1)	
	0	5	10	20
Number of tillers Number of panicles	10.7 b ^A 10.0 a	11.3 ab 10.3 a	12.0 ab 11.0 a	13.7 a 12.3 a
Number of grains per panicle	20.2 a	22.8 a	24.5 a	31.0 a
Dry weight of grains per panicle (g)	0.41 a	0.46 a	0.49 a	0.63 a
Dry weight of 100 grains (g)	2.01 a	2.02 a	2.02 a	2.02 a
Percentage of empty grain (%)	15.4 a	10.8 ab	10.8 ab	9.0 b
Harvésť index	0.19 a	0.21 a	0.21 a	0.25 a

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

S rate (kg ha ⁻¹)	ES (mg pot ⁻¹)	OS (mg pot ⁻¹)	Recovery c ES (%)	of fertiliser S OS (%)	
0 5 10 20	3.1 a ^A 3.2 a 3.4 a 4.1 a	394.5 a 405.7 a 408.2 a 413.8 a	0.8 a 1.4 a 2.1 a	32.0 a 36.8 a 51.3 a	

3.22 The residual effect of S-application rate on extractable S (ES), organic S (OS) and the recovery of fertiliser S in these fractions under non-flooded conditions.

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.23 The residual effects of different S sources on yield components under non-flooded conditions.

Source of S	No. of tillers	No. of panicles	Dw. of grains panicle ⁻¹ (g)	Dw. of a 100 grain (g)	percentage of empty grain (%)	Harvest index
G	12.0 bc ^A	11.0 ab	0.49 a	2.02 a	10.8 b	0.21 a
PVA	12.3 a-c	11.0 ab	0.59 a	2.02 a	10.3 b	0.23 a
LS	12.7 a-c	11.3 ab	0.55 a	2.01 a	10.0 b	0.22 a
HF	13.7 ab	12.3 a	0.62 a	2.00 a	10.1 b	0.23 a
SW	13.3 a-c	11.7 ab	0.60 a	2.03 a	10.7 b	0.23 a
US	13.0 a-c	11.3 ab	0.57 a	2.03 a	11.3 b	0.22 a
SCU	12.3 a-c	11.0 a	0 <i>.</i> 63 a	2.02 a	10.2 b	0.23 a
E	12.3 a-c	11.0 ab	0.60 a	2.03 a	10.3 b	0.24 a
SB	15.0 a	13.0 a	0.65 a	2.03 a	9.0 b	0.23 a
С	10.7 c	10.0 b	0.41 a	2.01 a	15.4 a	0.19 a

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

3.24 The residual effects of different S sources on extractable S (ES) and organic S (OS) under non-flooded conditions.

Source	ES	OS
of S	(mg pot ⁻¹)	(mg pot ⁻¹)
 С	3.1 c	394.5 a
G	3.4 bc ^A	408.2 a
PVA	3.6 bc	404.4 a
LS	3.5 bc	407.3 a
HF	4.4 ab	407.7 a
SW	4.0 bc	408.6 a
US	3.9 bc	405.0 a
SCU	3.8 bc	410.8 a
E	3.5 bc	403.9 a
SB	5.2 a	419.2 a

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
S	*	*	*	-	*	*	*	*	*	*	*	*	*	-	-	*
Р	*	*	*	*	*	*	-	-	-	-	-	_	-	*	-	*
L	*	-	*	*	*	*	*	*	*	*	-	*	-	-	-	-
CS	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-		-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	-	*	-	*	*	*	*	*	*	-	-	-	*	-	-	*
SL	*	-	-	*	-	-	*	-	-	-	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

4.1 Main effects and interaction terms for the clover yield occurring with C, S, P and L factors when analysis was performed at each harvest.

= significant at 0.05.

4.2 Main effects and interaction terms for the grass yield occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	_	-	-	-	-	_	-	_	-	-	_	-
S	*	*	*	*	*	-	-	-	*	*	*	*	*	*	*	*
P	*	*	*	*	*	*	*	*	-	-	-	*	-	-	-	*
L	*	-	-	*	*	-	-	-	-	-	-	-	*	-	-	-
cs	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	-	*	*	-	-	*	-	-	-	~	-	-	-	-	-	*
SL	*	-	-	*	-	-	*	-	-	~	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

4.3 Main effects and interaction terms for the pasture yield occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Ś	*	*	*	-	*	*	*	*	*	*	*	*	*	*	*	*
C S P	*	*	*	*	*	*	-	-	-	-	-	-	*	-	-	*
l i.	*	-	*	*	*	*	*	*	*	*	*	*	*	*	-	*
L CS CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
ĊP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
L ČL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL SP	*	*	*	_	-	*	-	*	*	-	-	*	-	-	-	-
SL	*	-	-	*	-	-	-	-	-	-	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	_	-	-	-	-	-	-	-	-	-
ČŠL	-	-	-	-	-	-	-	-	-	-	-	-	-	_	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

* = significant at 0.05.

4.4 The effects of S and P sources on dry weight, S content, P content, percentage of S derived from the fertiliser and the recovery of fertiliser S in root under leached and non-leached conditions.

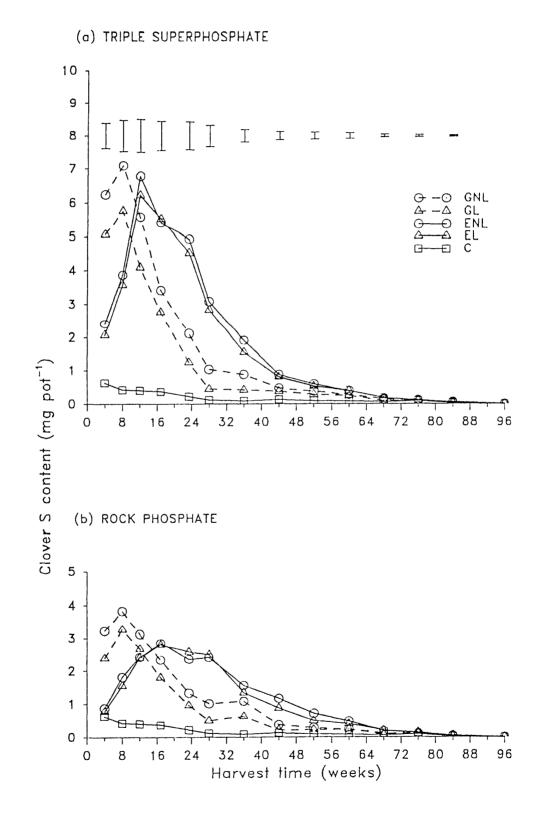
Treatment	Dry weight (g pot ⁻¹)	S content (mg pot ⁻¹)	P content (mg pot ⁻¹)	S derived from the fertiliser (%)	Recovery of fertiliser S (%)
TSP ENL EL GNL GL RP	11.2 a ^A 10.2 ab 8.5 bc 8.2 bc	12.6 a 11.0 a-c 9.4 a-c 8.1 c	13.7 a 12.0 ab 11.9 ab 9.2 cd	11.0 ab 10.2 ab 7.7 c 7.4 c	3.6 a 2.8 a-c 1.8 bc 1.5 c
ENL EL GNL GL	8.7 bc 8.4 bc 8.1 bc 7.3 c	11.5 ab 10.2 a-c 9.3 bc 7.9 c	11.3 a-c 11.2 bc 10.9 bc 8.3 d	11.9 a 11.1 ab 8.9 bc 7.7 c	3.4 ab 2.9 a-c 2.1 a-c 1.5 c

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.5 Main effects and interaction terms for the clover S content occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	-	-	-	-	-	-	-	_	-	-	-	-
S	*	*	*	-	*	*	*	*	*	*	*	*	*	-	-	*
S P	*	*	*	*	*	*	-	-	-	-	-	_	-	*	-	*
	*	*	*	*	-	*	*	*	*	*	-	-	-	-	_	*
L CS CP CL SP SL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	*	*	-	*	*	*	-	*	*	-	-	-	-	-	-	*
SL	*	*	-	*	-	-		-	-	-	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	_	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

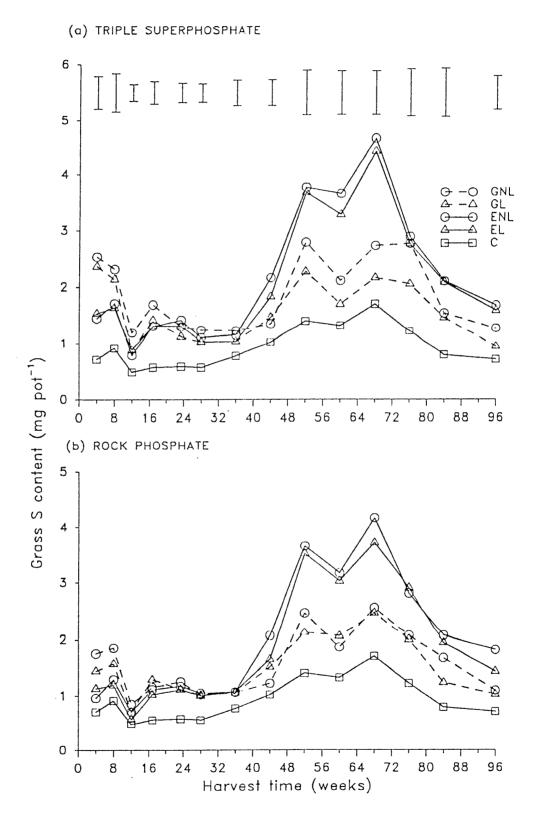
4.6 The effects of S and P sources on clover S content under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for $S \times P \times L$ interaction at each harvest.



Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	-	-	-	-		-	_	_	-	_	_	-
S	*	*	*	*	*	-	-	-	*	*	*	*	*	*	*	*
Р	*	*	*	*	*	-	-	-	-	-	-	*	-	-	-	*
L	*	-	-	-	-	-	-	-	-	-	-	*	-	-	*	-
CS	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	-	-	-	-	-	-	-	-	-	-	-	*	-	-	-	-
SL	-	-	-	-	-	-	-	-	*	-	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	_	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	_
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

4.7 Main effects and interaction terms for the grass S content occurring with C, S, P and L factors when analysis was performed at each harvest.

4.8 The effects of S and P sources on grass S content under leached and non-leached conditions. Vertical bars indicate LSD (P = 0.05) for $S \times P \times L$ interaction at each harvest.



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Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S imes T
С	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
S	*	*	*	-	*	*	*	*	*	*	*	*	*	*	*	*
Р	*	*	*	*	*	*	-	-	-	-	-	-	-	-	-	*
L	*	*	*	*	-	*	*	*	*	*	-	*	-	-	*	-
CS	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
СР	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	*	*	-	*	*	*	-	-	-	-	-	-	-	-	-	*
SL	*	*	-	*	*	-	*	-	-	-	-	-	-	-	-	-
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

4.9 Main effects and interaction terms for the pasture S content occurring with C, S, P and L factors when analysis was performed at each harvest.

* = significant at 0.05.

4.10 Main effects and interaction terms for the pasture P content occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	0 - 17	17 - 36	36 - 68	68 - 96	Total
С	-	-	-		-
S	*	*	*	*	*
P	*	*	*	*	*
L	*	-	-	*	*
CS	-	-	-	-	-
CP	-	-	-	-	-
CL	-	-	-	-	-
SP	-	-	*	-	*
SL	-	-	-	-	*
PL	-	-	-	-	-
CSP	-	-	-	-	-
CSL	-	-	-	-	-
CPL	-	-	-	-	-
SPL	-	-	-	-	-
CSPL	-	-	-	-	-

4.11 Main effects and interaction terms for the percentage of S in pastures derived from the fertiliser occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	All	4	8	36	68	96	S imes T
С	-	-	-	_	-	-	-
S	*	*	*	*	*	*	*
Р	*	*	*	*	-	-	*
L	*		-	*	*	-	*
CS	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-
SP	*	*	*	*	-	-	*
SL	-	-	-	-	-	-	*
PL	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-
SPL	-	-	-	-	*	-	-
CSPL	-	-	-	-	-	-	-

* = significant at 0.05.

4.12 Main effects and interaction terms for the recovery of fertiliser S in pastures occurring with C, S, P and L factors when analysis was performed at each harvest.

Source	All	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	_	-	-	_	-	-	-	-	-	-	-	_	-
S	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
Ρ	*	*	*	*	*	-	*	-	-	-	-	-	-	-	-	*
L	*	*	*	*	*	*	*	*	*	*	*	*	-	-	-	*
CS	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	*	-	*	*	*	*	-	-	-	-	-	-	-	-	-	*
SL	*	*	-	*	-	*	*	-	-	-	-	-	-	-	-	*
PL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSPL	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

Source	0 - 17	17 - 36	36 - 68	68 - 96	Total
С	-	-	-	-	_
S	*	*	-	-	*
Р	*	*	-	-	-
CS	-	-	-	-	-
CP	-	-	-	-	-
SP	-	-	-	-	-
CSP	-	-	-	-	-

4.13 Main effects and interaction terms for the S content of leachate occurring with C, S and P factors when analysis was performed at each harvest.

* = significant at 0.05

4.14 The effects of S and P sources on the S content of leachate (mg pot ⁻¹) at each observation time.

Treatment	0 - 17	17 - 36	36 - 68	68 - 96	Total
TSP					
E	3.90 c ^A	3.30 c	4.63 a	3.88 a	15.72 b
G	8.89 a	4.30 ab	4.56 a	3.75 a	21.5 a
RP					
E	3.20 c	3.77 bc	4.96 a	4.19 a	16.11 b
G	7.79 b	4.56 a	4.73 a	3.87 a	20.95 a

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.15 Main effects and interaction terms for the P content of leachate occurring with C, S and P factors when analysis was performed at each harvest.

Source	0 - 17	17 - 36	36 - 68	68 - 96	Total
С	-	-	-	-	-
S	-	-	-	-	-
P	*	-	-	-	*
CS	-	-	-	-	-
CP	-	-	-	-	-
SP	-	-	-	-	-
CSP	-	-	-	-	-

4.16	The effects of S and P sources on the P content of leachate (mg pot -1)	
	at each observation time.	

Treatment	0 - 17	17 - 36	36 - 68	68 - 96	Total
TSP	1.16 a ^A	0.44 a	0.42 a	0.34 a	2.37 a
RP	0.63 b	0.45 a	0.44 a	0.39 a	1.9 b

^A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.17 Main effects and interaction terms for the percentage of S in leachates derived from the fertiliser occurring with C, S and P factors when analysis was performed at each harvest.

Source	All	4	8	36	68	96	$S\timesT$
С	-	-	-	-	-	-	-
S	*	*	*	*	*	*	*
Р	*	*	-	-	-	-	*
CS	-	-	-	-	-	-	-
СР	-	-	-	-	-	-	-
SP	*	*	-	-	*	-	*
CSP	-	-	-	-	-	-	-

= significant at 0.05.

4.18 Main effects and interaction terms for the recovery of fertiliser S in leachates occurring with C, S and P factors when analysis was performed at each harvest.

Source	Ali	4	8	12	17	24	28	36	44	52	60	68	76	84	96	S×T
С	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
S	*	*	*	*	*	-	-	-	-	-	-	-	-	-	-	*
P	*	*	*	*	-	-	-	-	-	-	-	-	-	-	-	*
CS	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SP	-	*	-	*	-	-	-	-	-	-	-	-	-	-	-	*
CSP	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

4.19 The effects of S and P sources on extractable S (ES), organic S (OS), percentage of S derived from the fertiliser and the recovery of the fertiliser S in these fractions under leached and non-leached conditions.

Treatment	ES (mg pot ⁻¹)	S derived from the fertiliser (%)	Recovery of the fertiliser S (%)	OS (mg pot ⁻¹)	S derived from the fertiliser (%)	Recovery of the fertiliser S (%)
TSP						
ENL	17.4 a ^A	16.0 a	6.8 a	199.6 a	5.2 b	25.3 c
EL	16.7 a	13.1 a	5.6 a	178.8 a	3.1 c	13.6 d
GNL	13.5 a	13.9 a	5.6 a	204.8 a	4.9 b	24.3 c
GL	13.2 a	12.8 a	4.4 a	153.9 a	2.8 c	10.2 e
RP						
ENL	19.1 a	14.2 a	6.6 a	191.7 a	11.2 a	50.8 a
EL	15.4 a	11.7 a	4.6 a	148.0 a	10.2 a	36.7 b
GNL	15.2 a	12.2 a	4.7 a	175.3 a	11.2 a	47.7 a
GL	14.9 a	12.1 a	4.7 a	189.7 a	6.7 b	31.0 b

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.20 The effects of S and P sources on total P content (mg pot-1) of soil under leached and non-leached conditions.

Treatment	Total P (mg pot ⁻¹)	
TSP ENL EL GNL GL	242.8 a ^A 217.6 a 248.0 a 207.0 a	
RP ENL EL GNL GL	231.7 a 200.0 a 230.8 a 213.2 a	

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.21 The effects of different S sources on extractable S (ES), organic S (OS), percentage of S derived from the fertiliser and recovery of the fertiliser S in these fractions.

ES Treatment (mg pot ⁻¹)	S derived from the fertiliser (%)	Recovery of the fertiliser S (%)	OS (mg pot ⁻¹)	S derived from the fertiliser (%)	Recovery of the fertiliser S (%)
G 12.6 a ^A PVA 16.3 a SW 18.8 a HF 20.9 a LS 17.6 a C 14.5 a	13.4 a 15.6 a 16.1 a 16.5 a 12.5 a	4.2 b 5.7 ab 7.1 ab 8.7 a 5.3 ab	212.9 a 212.2 a 229.2 a 228.1 a 226.3 a 164.2 a	4.7 a 4.9 a 5.6 a 5.8 a 4.5 a	25.0 a 24.9 a 32.0 a 33.5 a 25.3 a

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).

4.22 The effect of different S sources on total P content (mg pot-1) of soil.

Treatment	Total P (mg pot ⁻¹)	
G	258.9 a ^A	
PVA SW HF LS	258.2 a 262.8 a 267.0 a 237.8 a	
с	199.8 a	

A Data followed by the same letter in the same column are not significantly different (P < 0.05) (Duncan's multiple range test).