Study of Sulphur Fractions in Calcareous Soil by using different Sulphur Sources and Grades of Sulphur

Abstract

An incubation experiment was conducted in the department of Crop management, TRIARD- Roever Agriculture College, Perambalur to study the S release pattern by different S sources and grades. The bulk soils were collected from the Esanai village which has deficient in sulphur. The treatments were replicated thrice in completely randomized design. Treatment details are Ammonium sulphate, Single Super Phosphate, Gypsum were the three sources of sulphur applied at the intervals of 0, 20, 40 kg S ha^{-1} along with the recommended dose of N, P and K. Destructive soil sampling was carried out at 15, 30, 60, 90 days after incubation and analyzed for S fractions as per standard procedures to understand the effect of S sources and S dynamics in those areas. Soil samples were analysed for sulphur fraction *i.e.* Water soluble sulphur fraction, Exchangeable sulphur fraction, $CaCl_2$extractable sulphur fraction, Occuled sulphur fraction, Organic sulphur fraction and Total sulphur fraction. The soil sulphur fractions were arranged in the order of occluded sulphur > water soluble sulphur > exchangeable sulphur > CaCl2 extractable sulphur > organic sulphur. The results emerged out in this experiment indicate that that application of sulphur in the form of Ammonium Sulphate @ 40 kg S ha^{-1} was found to be applied for better release of available sulphur. The zero fertilization led to decline in the levels of all the forms of sulphur as compared to rest of treatments.

Keywords : Sulphur fraction, Ammonium sulphate, calcareous soil

Introduction

Among the major nutrients, sulphur as an essential nutrient to the plants. It also plays an important role in constituent of the proteinaceous amino acids such as methionine and cysteine, glutathione, vitamins (biotin and thiamine), phytochelatins, chlorophyll, coenzymes A and S-adenosyl – methionine (Nakai *et al.,* 2021). In the absence of sufficient sulphur, several essential enzymatic activities and physiological functions are inhibited. Thus maintenance of an optimum level of sulphur in the soil is essential for maximum crop production and its quality aspects. Sulphur exists in soil as free and adsorbed sulphate and in diverse organic and inorganic compounds. In the humid region it is in the organic forms, while in arid soils the sulphate salts of calcium, magnesium, sodium and even potassium predominate (Kanwar, 1976). Incidental sulphur returns to soil is possible through farmyard manure and the use of conventional sulphur containing fertilizers, such as Ammonium sulphate (24 per cent S), Ammonium phosphate sulphate (14 per cent S), Elemental Sulphur (90-99 per cent), Gypsum (17 per cent S) and Potassium sulphate (12 per cent S). Before fertilizing, it is necessary to understand the knowledge of different forms of sulphur and factors affecting their distributions are essential in improving the sulphur nutrition of the crops.

Materials and Methods

An incubation experiments was conducted in the department of Crop Management, Thanthai Roever Institute of Agricultural and Rural Development, Perambalur during May 2022 to study the S release pattern by different S sources and grades of S. Eight treatments were laid out in CRD with 3 replications. The treatments from T_1 to T_8 are absolute control, RDF alone, Gypsum @ 20 and 40 kg S, SSP @ 20 and 40 kg S and Ammonium sulphate @ 20 and 40 kg S. The bulk soils were collected from the surface layer (0-15cm) of soils situated in onion growing areas of Esanai was used for this experiment which was deficient in S. The plastic containers filled with 100 grams of processed soils with adequate quantity of water to maintain field capacity. Destructive soil sampling was carried out at 15, 30, 60, and 90 days after incubation and analysed for S fractions. The prepared soil samples were analyzed for determining fraction of sulphur by adopting standard methods (Chesnin and Yien, 1950) as described below. The total sulphur was estimated using the method followed by Hesse, 1971. The results were statistically analysed using Aggress software at 5% level of significance.

Estimation of Sulphur Fractions

Water soluble sulphur

Soil sample was extracted with distilled water 1:10 (w/v) ratio for 30 minutes in mechanical shaker. Then it was centrifuged for ten minutes at 10000 rpm. 5 ml of the supernatant was pipetted out in 25 ml volumetric flask. 10 ml of sodium acetate- acetic acid buffer (pH 4.8) was added followed by 1 ml of gum acacia solution and 1 g of $BaCl_2$. The volume was made up to 25 ml with distilled water and the reading was taken in UV-VIS spectrophotometer at 420 nm wavelengths after adjusting the meter to zero per unit absorbance with the blank.

Exchangeable or adsorbed sulphur

After the centrifugation process, samples were extracted with 0.032 M NaH_2PO_4 at 1:10 (w/v) ratio after centrifugation. Following 30 minutes of shaking, each sample was again centrifuged at 10,000 rpm for about 10 minutes. The extracted supernatant was used to estimate S by turbidimetry method.

Occluded/ Precipitated sulphur

The residue of the previous fraction was extracted with 1M HCl with the ratio of 1:20 (w/v). Followed by 60 minutes of shaking, the samples were centrifuged for about 10 minutes at 10,000 rpm. The extracted S was estimated by using turbidimetry method.

Organic Sulphur

One gram of air dried (20 mesh) soil along with 1 g of $NaHCO_3$ was taken in a silica crucible. The mixture was placed in a muffle furnace for 3 hours at 500° C. After cooling, the contents were transferred to a 100 ml conical flask to which 25 ml of extracting solution (Dissolve 4.6 g of NaH_2PO_4. H_2O in 1 L of 2 N acetic acid) was added. After the reaction subsides, the contents of the flask were shaken for half an hour. Later the solution was filtered through a dry Whatman No. 1 filter paper. An aliquot of the filtrate was taken for the turbidimetrical determination of sulphate.

Total Sulphur

20 g of soil sample was taken in the silica crucible and ignited in the muffle furnace and was transferred to 250 ml conical flask. 60 ml of 1:1 HCl was added to the conical flask and kept in the sand digester until the solution turned colourless. At the end of digestion, the contents in the flask were diluted with 200 ml of distilled water and it was filtered through Whatman No. 3 filter paper and the filtrate was collected in 500 ml volumetric flask. The entire contents were washed with hot water by transferring into the filter paper until free of chloride was achieved. After free of chloride, the contents were cooled and the volume was made up to 500 ml with distilled water. 50 ml of sesquioxide extract was pipetted out in 500 ml beaker and 2 g of solid ammonium chloride was added for crystallization. The solution was heated and 10 ml of 10% barium chloride was added drop by drop with constant stirring and the boiling was continued for another 3 minutes. The beaker was kept in a sand bath for 30 minutes to promote granulation. After granulation, the contents were washed with hot water by transferring to Whatman No. 42 filter paper till free of chloride was achieved. The filter paper along with the precipitate was added to the weighed silica basin and dried in the oven. It was ignited over a low flame initially and the filter paper was made to ash completely. The weight of the barium sulphate obtained was considered as the sulphate content of the sample (Hesse, 1971).

Results and Discussion

Initial Physico-Chemical Characteristics of Soil

The pH of the experimental soil was slightly alkaline in reaction (8.03). The soil was free from salinity with an electrical conductivity of 0.20 dS m^{-1}. The soil belonged to sandy clay loam in texture. The major nutrient status of the soils showed that low available N content (179 kg ha^{-1}), the high status of available P and Medium K status (78 and 189 kg ha^{-1} respectively) The soil contains low available S (9.2 mg kg^{-1}).

Sulphur Fractions

The sequential extraction of various S fractions was determined and the data is presented in Tables 1 to 3. Among the different fractions, the water soluble contribution was found to be low followed by Exchangeable, $CaCl_2$ extractable, occluded and organic S. The contribution of total- S was the highest among all other zinc fractions studied.

Water Soluble S Fraction

The water soluble sulphur fractions of soil ranged from 6.75 mg kg^{-1} in control to 18.33 mg/kg in AS @ 40kg S ha^{-1}. The highest value of water soluble sulphur in soil was registered with the application of AS @ 40 kg S ha^{-1} (15.45, 16.45, 18.33, 16.22 mg kg^{-1}) which was also on par with application of SSP @ 40 kg S ha^{-1} (14.5, 15.95, 17.25, 15.43 mg kg^{-1}) at 15, 30, 60 and 90 DAI, respectively (Table 1). The releasing pattern of water soluble sulphur from soil had increased till 60 DAI and then slightly decreased up to 90 DAI for all the sources and the levels of sulphur applied. But, the trend seemed to be in contrast to treatments with the control and RDF alone applied lots which where decreasing throughout the period of incubation. These results have been ascribed to finding of Rajani *et al.* (2010) that the omission of S in the fertilizer has caused a continuous depletion in the available S status of soil.

Exchangeable S Fraction

The soil exchangeable sulphur fraction ranged from 5.45 mg kg^{-1} in control to 12.57 mg kg^{-1} in AS @ 40 kg S ha $^{-1}$ (Table 1). The highest value of soil exchangeable sulphur was recorded with the application of AS @ 40 kg S ha^{-1} (10.97, 11.19, 11.87, 12.21 mg kg^{-1}) and followed by application of SSP @ 40 kg S ha $^{-1}$ (10.5, 10.53, 11.54, 11.13 mg kg^{-1}). The releasing pattern of exchangeable sulphur from soil had increased till 60 DAI and then slightly decreased at 90 DAI for all the sources and levels of sulphur applied. Higher S release in AS treated cups might be due to relatively higher solubility of AS as compared to SSP and gypsum (Scherer, 2001)

CaCl$_2$ Extractable S Fraction

The soil CaCl$_2$ extractable sulphur fraction ranged from 97.11 mg kg^{-1} in control to 27.84 mg kg^{-1} in AS @ 40 kg S ha^{-1}. The highest value of soil CaCl$_2$ extractable sulphur was observed with the application of AS @ 40 kg S ha^{-1} (26.22, 27.58, 29.75, 32.55 mg kg^{-1}) which was also on par with the application of gypsum @ 40 kg S ha^{-1} (24.52, 25.15, 27.04, and 29.63mg kg^{-1}) (Table 2). The lowest value of soil CaCl$_2$ extractable sulphur in soil was observed with control (9.0, 8.53, 8.22, 7.11 mg kg^{-1}). The soil CaCl$_2$ extractable sulphur fractions had shown a constant increase throughout the period of incubation for all the sources and levels of sulphur applied. The similar findings were found in Nivetha *et al.,* 2019.

Occluded S Fraction

The soil occluded sulphur fraction ranged from 32.21 mg kg^{-1} in control to 67.50 mg kg^{-1} (Table 2). The highest value of soil available sulphur was observed with the application of AS @ 40 kg S ha^{-1} (67.74, 65.90, 67.50 and 66.25 mg kg^{-1}) and followed by application of SSP @ 40 kg S ha^{-1} (61.12, 61.50, 63.40 and 62.45 mg kg^{-1}). The mean values of occluded sulphur were in line with the findings of Sankaran, 1989.

Organic S Fraction

Organic sulphur was one of the dominant fraction of sulphur. It was ranged from 52.7 mg kg^{-1} in RDF alone to 160.07 mg kg^{-1} in AS @ 20 kg S ha^{-1} (Table 3). The trend seemed to be constantly decreasing throughout the period of incubation for all the sources and levels of sulphur applied. The higher amount of organic sulphur was registered with the application of AS @ 20 kg S ha^{-1} (160.07, 159.23, 156.2 and 152.22 mg kg^{-1}) and the lowest values were observed with control (54.16, 56.28, 60.64 and 65.73 mg kg^{-1}). Similar results were reported by Singh *et al.* (2001) that the buildup in S status was mostly through organic sulphur fraction.

Total S Fractions

The soil total sulphur ranged from 110.57 mg kg^{-1} in control to 241.01 mg kg^{-1} in AS @ 40 kg S ha^{-1} (Table 3). The studies on total sulphur revealed that, all the sources and levels of sulphur applied were on par and superior to the control and RDF alone throughout the period of incubation. Setia and Sharma (2005) that the amount of all the fraction of S increased with the application of AS @ 40 kg S ha^{-1}.

Conclusion

The results can be summarized that application of sulphur in the form of Ammonium Sulphate @ 40 kg S ha^{-1} was found to be applied for better release of available sulphur. The zero fertilization led to decline in the levels of all the forms of sulphur as compared to rest of treatments.

Table 1: Effect of sources and levels of sulphur on soil water soluble sulphur and Exchangeable sulphur fraction (mg kg^{-1})

Treatments	**Water Soluble Sulphur (mg kg^{-1})**				**Exchangeable Sulphur (mg kg^{-1})**			
	15 DAI	**30 DAI**	**60 DAI**	**90 DAI**	**15 DAI**	**30 DAI**	**60 DAI**	**90 DAI**
T_1	6.75	6.23	4.73	3.08	5.45	5.21	4.22	3.56
T_2	7.75	7.89	9.87	6.91	6.04	6.81	7.45	7.14
T_3	13.75	14.75	15.5	12.42	10.15	10.32	11.21	11.27
T_4	15.45	16.45	18.33	16.22	10.97	11.19	11.87	12.21
T_5	11.85	12.5	11.75	10.33	8.25	8.12	9.69	10.42
T_6	14.5	15.95	17.25	15.43	10.5	10.53	11.54	11.13
T_7	10.35	10.35	11.67	9.07	7.5	7.75	8.57	9.15
T_8	12.75	13.25	14.13	11.75	9.35	9.78	10.63	10.98
Mean	11.64	12.17	12.90	10.65	8.52	8.71	9.39	9.48
CD (p=0.05)	1.26**	1.30**	1.41*	0.72**	1.07**	1.09*	0.67*	0.64*
SEd	0.68	0.72	0.77	0.66	0.49	0.50	0.54	0..54

Table 2: Effect of sources and levels of sulphur on soil $CaCl_2$ extractable sulphur fraction (mg kg^{-1}) and Occluded Sulphur (mg kg^{-1})

Treatments	**$CaCl_2$ Extractable Sulphur (mg kg^{-1})**				**Occluded Sulphur (mg kg^{-1})**			
	15 DAI	**30 DAI**	**60 DAI**	**90DAI**	**15 DAI**	**30 DAI**	**60 DAI**	**90 DAI**
T_1	9.00	8.53	8.22	7.11	35.21	34.86	33.70	32.50

T_2	9.43	8.23	8.49	7.35	36.65	36.32	35.19	34.21
T_3	19.05	20.62	22.23	25.98	58.35	58.89	60.41	59.40
T_4	26.22	27.58	29.75	32.55	65.74	65.90	67.50	66.35
T_5	21.23	23.33	24.69	26.22	49.25	49.69	51.79	50.59
T_6	22.04	23.11	25.72	27.84	61.12	61.50	63.40	62.45
T_7	18.15	19.17	21.42	23.96	45.67	45.89	47.79	46.49
T_8	24.52	25.15	27.04	29.63	53.12	53.65	55.43	54.81
Mean	18.70	19.46	20.94	22.58	50.63	50.83	55.27	50.85
CD(p=0.05)	0.90**	0.85*	0.85*	0.79**	1.91*	1.87*	1.89*	1.83**
SEd	1.11	1.16	1.26	1.37	2.86	2.90	2.91	2.92

Table 3: Effect of sources and levels of sulphur on soil Organic Sulphur and Total Sulphur fraction (mg kg^{-1})

Treatments	**Organic Sulphur (mg kg^{-1})**				**Total Sulphur Fraction (mg kg^{-1})**			
	15 DAI	**30 DAI**	**60 DAI**	**90 DAI**	**15 DAI**	**30 DAI**	**60 DAI**	**90 DAI**
T_1	54.1	56.2	63.4	65.7	110.57	110.84	111.57	112.05
T_2	52.7	54.4	59.34	61.9	110.85	111.01	112.81	113.93
T_3	160.0	159.2	154.8	152.2	235.4	235.56	235.98	238.22
T_4	155.6	154.5	147.2	147.2	238.45	238.91	239.89	241.01
T_5	130.1	128.3	122.7	120.3	227.08	227.27	227.66	228.59
T_6	125.9	122.5	116.9	114.4	229.21	229.41	229.77	230.32
T_7	140.3	139.3	134.0	132.8	231.09	231.39	231.83	232.28
T_8	137.9	135.0	129.9	128.3	233.08	233.56	233.86	234.89
Mean	119.6	118.6	116.2	115.3	201.96	202.24	202.92	203.91
CD(p=0.05)	2.41*	2.46*	4.41*	2.66*	3.47*	5.63*	3.49*	3.50*

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