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Sulphur Transformation in Acid Soils under Closed Incubation Studies

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ABSTRACT: A laboratory investigation was conducted with five levels of Sulphur (0, 10, 20, 30 and 40 (mg S kg^{-1}) to study changes in different extractants of sulphur under field capacity moisture regime. Release of available sulphur in soils was evaluated under application of gypsum as sulphur source. Soils were sampled and analysed on 30, 60 and 90th day of incubation period. Results revealed that application of gypsum @ 30 mg S kg^{-1} lead to accumulation of higher amount of soluble sulphur. Sulphur in the soils were extracted with four different extractants *i.e.*, 500 ppm $Ca(H_2PO_4)_2.H_2O$, 0.5M $NaHCO_3$, 0.5M NH_4OAc and 0.15% $CaCl_2$. An attempt was also made to explain the variation in sulphur mineralization through soils correlations between the amounts of mineralised soil and some individual sulphur extractant. The soil samples were drawn on 30th days interval to monitor changes in different extractants of sulphur. The experiment was set up in a completely randomized design with five treatments and four replications. The levels had a significant effect on releasing pattern of S at different days of incubation. The result indicated that the releasing pattern of S was high when increasing the incubation time and increased the S level. Among the different levels of sulphur, S@ 30 mg S kg^{-1} was extracted more sulphur from the soil. The significant correlation between different extractants of sulphur suggested an interrelated dynamic equilibrium amongs all extractants of sulphur.

Keywords: Sulphur, Incubation, Extractants, Gypsum, Field Capacity.

INTRODUCTION

FAO (2021) reported global rice production in 2021 is still seen around 518 million tonnes (milled basis), up 0.9 % year-on-year and a fresh peak. The latest FAO forecast of rice stocks at the close of the 2021/22 marketing seasons has undergone only minor adjustments since November and is still seen hovering around a record of 188 million tonnes. World rice trade in 2022 (January-December) is predicted to reach 51.4 million tonnes, up 4.9 % from the expected level for 2021. Abundant supplies are still expected to sustain a 1.6 % annual expansion in global rice utilization in 2021/2022 to an all-time high of 519 million tonnes. Agriculture is the mainstay of the Indian economy, contributing about 23 % of gross domestic product (GDP) and providing a livelihood to two-thirds of the population. The net cultivated area has been about 141 million ha for the last 30 years. However, there has been a progressive increase in the gross cropped area as the cropping intensity has increased from 118 to 135 % in the last three decades. The total gross cropped area is about 190 million ha (Anonymous, 2019). In India, according to the first estimates released by the agriculture and farmers welfare ministry, the kharif rice production is expected to reach a record level of 107.04 mt during 2021-2022, which is slightly higher than last year's figure of 104.41 mt. (Sutanuka, 2021). As crop demands for S increase, deficiencies are more likely to occur on Soils that inherently supply less available S within rooting zone. Minimum use of low- analysis fertilizers like ammonium sulphate and single super phosphate and organic manures has rendered the Indian soils deficient in sulphur. Sulphur is one of the most limiting nutrients for agricultural production in many Asian countries creates S deficiency in soils due to continuous cropping and regular use of S free fertilizers in several agro-ecological zones. Continuous removal of S from soils by plant uptake has led wide spread S deficiency and soil S budget all over the world (Aulakh *et al.***,** 1977). Role of sulphur in Indian agriculture is now gaining importance because of the recognition of its role in increasing crop production, not only of oil seeds, pulses and forages but also of many cereals (Singh *et al*., 2000). Sulphur deficiency in crops is gradually becoming widespread due to continuous use of sulphur free fertilizers, high yielding crop varieties, intensive multiple cropping systems coupled with higher productivity. Sulphur is essential for protein formation, important for high protein content, a component of vitamin A and activates certain enzyme systems in plants (Havlin *et al*., 2004). Sulphur is best known for its role in the synthesis of proteins, oils, vitamins and flavoured compounds in plants. About 90% of sulphur is present in amino acids (Somnath and Goutam 2012).

MATERIALS AND METHODS

A total of thirty bulk soil samples (0-15 cm) from three blocks of Imphal East District, Manipur having different physico-chemical properties were collected under stratified random sampling – proportional allocation method. The collected soil samples were then allowed to air dried in shade, ground with wooden mortar and pestle and passed through a 2 mm sieve. The sieved soil samples has been stored in labeled polythene bags for determination of different soil parameters and incubation studies**.** The climate of Manipur falls under sub-tropical humid type with mild, dry winters and a hot monsoon season. An incubation study was carried out to determine sulfur transformation/mineralization rates in soil samples obtained from representative soils of Imphal East District, Manipur. The experiment was conducted to study the release pattern of sulphur in different soils of Manipur. From the thirty soil samples five samples were selected for incubation studies. 30 g of 2 mm sieved soil sample was filled with 250 ml (depth 9 cm, diameter 21cm) plastic containers. The soil has been incubated at room temperature for 90th days at field capacity by applying water as and when required. The loss water from the samples were checked by weighing the sample at 3 days interval and accounted only for 0.2-0.3 ml of water in 1 week. The soil samples were drawn on 30, 60 and 90th days interval after incubation to monitor changes in different extractants of sulphur. The soil available sulphur were extracted with solution of 500ppm $Ca(H_2PO_4)_2.H_2O$ (Ensminger, 1954), 0.5M NH_4OAc (Rehm and Caidwell, 1968), 0.15% $CaCl_2$ (Williams and Steinbergs 1959) and 0.5M $NaHCO_3$ (Victor and Nearpass 1960). The soil samples were shaken for half an hour with a soil to solution ratio of 1:5 and centrifuged and extractable S were determined turbidimetrically (Chesnin and Yien 1951). The experiment was set up in a completely randomized design with five treatments and four replications. The treatments were: T_0 (Control), T_1 (10 mg S kg^{-1}), T_2 (20 mg S kg^{-1}), T_3 (30 mg S kg^{-1}) and T_4 (40 mg S kg^{-1}). Gypsum was used as a source of sulphur.

RESULT AND DISCUSSION

The physical and chemical characteristics of bulk soil samples are presented in Table 1. The sand, silt and clay fractions varied from 8.20 - 34.70 %, 6.10 – 32.50 % and 36.40 – 80.92 %. Majority of the soils were clay in texture. The pH of the soils varied from 4.59 – 5.43 with a mean value of 5.03. The EC content of the soils varied from 0.05 – 0.29 d Sm^{-1} with a mean value of 0.13 d Sm^{-1}. The organic carbon content of the soil ranging from 10.5 – 27.0 g kg^{-1} with a mean value of 17.2 g kg^{-1}. CEC of the soils ranged from 10.28 – 20.10 Cmol (p+) kg^{-1} with a mean value of 15.07 Cmol (p+) kg^{-1}. The available N, P and K ranged from 214.21 – 489.85 Kg ha^{-1}, 17.28 – 57.62 Kg ha^{-1} and 145.89 – 369.47 Kg ha^{-1} with a mean values of 330.22 Kg ha^{-1}, 28.80 Kg ha^{-1} and 266.08 Kg ha^{-1}, respectively. The exchangeable Ca^{2+} and Mg^{2+} of the soil varied from 0.64-1.95Cmol (p+) kg^{-1} and 0.44-1.03Cmol (p+) kg^{-1} with a mean values of 1.31Cmol (p+) kg^{-1} and 0.75Cmol (p+) kg^{-1}. Kher and Singh (1993) and Patel *et al*. (2011) indicating that these soil properties played a major role in availability of sulphur content.

Incubation Studies. The data pertaining to effect of different levels of sulphur on 500 ppm Monocalcium Phosphate ($Ca\ (H_2PO_4)_2.H_2O$) under incubation are presented in Table 2. As the period of incubation advanced, a continuous increase in 500 ppm Monocalcium Phosphate content of soils was noticed in 30, 60 and 90th days of incubation (DOI). The data on 500 ppm Monocalcium Phosphate extractant indicated significant difference between treatments. The highest available sulphur content was observed on the 90th day of incubation. At 30, 60 and 90th DOI, the highest 500 ppm Monocalcium Phosphate extractant content of 51.76, 60.27 and 67.34 mg S kg^{-1} was noticed in the treatment of T_3 – 30 mg S kg^{-1}. It was on par with the treatment received T_4 – 40 mg S kg^{-1}. Similar with the finding of Luxmi *et al*. (2018). Aluminium and iron oxides are the two major components involved in sulphate adsorption by soils. When 500 ppm MCP solutions are introduced to the soil, Phosphate ions displace SO_4^{2-} ions from retention sites into the solution. The superiority of 500 ppm MCP can be explained by the phosphate ion having greater power to displace the adsorbed sulphate than does the acetate ion, determining higher values of extractable sulphate, Aylmore *et al*. (1967).

Table 1: Particle size distribution of the soils of Imphal East District covering all blocks.

Soil properties	Range	Mean
Sand (%)	8.20 - 34.70	25.60
Silt (%)	6.10 - 32.50	18.20
Clay (%)	36.40 – 80.92	56.78
pH	4.59 – 5.43	5.03
EC (dSm^{-1})	0.05 – 0.29	0.13
OC (g kg^{-1})	10.5 – 27.0	17.2
CEC (Cmol(p+) kg^{-1})	10.28 – 20.10	15.07
Available N (Kg ha^{-1})	214.21 – 489.85	330.22
Available P (Kg ha^{-1})	17.28 – 57.62	28.80
Available K (Kg ha^{-1})	145.89 – 369.47	266.08
Exchangeable Ca^{2+} (Cmol(p+) kg^{-1})	0.64 - 1.95	1.31
Exchangeable Mg^{2+} (Cmol(p+) kg^{-1})	0.44 -1.03	0.75

Among the treatments, the highest 0.5M NH_4OAc (Ammonium acetate) content of 39.23 , 44.28 and 49.03 mg S kg^{-1} at 30, 60 and 90th DOI was noticed in the treatment that received 30 mg S kg^{-1} (T_3) and it was on par with the treatments of 40 mg S kg^{-1} (T_4). As ammonium acetate acetic acid is an acidic reagaent, additionally, it solubilizes some fraction of inorganic sulphur compounds from soils (Rehm and Caldwell 1968). Several volatile organic S compounds are emitted from flooded soils. Among the compounds isolated in significant amounts are carbon disulfide, carbonyl sulfide, methyl mercaptan, dimethyl sulfide and dimethyl disulfide (Freney and Boonjawat 1983). At 30, 60 and 90th DOI, the highest 0.15% $CaCl_2$ (Calcium chloride) content (25.09, 32.62 and 36.29 mg S kg^{-1}) was recorded in the treatment that received 30 mg S kg^{-1} (T_3) and it was on par with the treatment of 40 mg S kg^{-1} (T_4). The higher exchangeable Ca^{2+} led to increase the S adsorption by $CaSO_4$ bonding by the forces of chemisorption. These chemisorbed S was reversibly to release the labile pool by slow retention and also reactive surface of permanent clays would have increased the microbial load for S oxidation has improved the S availability 0.15% $CaCl_2$ extractant provided the best index of microbial mineralisation of S in soils. The increase in available S with increasing levels of applied S and time of incubation. Increase in the available sulphate S in the S treated acid soil is due to the multiplication of S oxidizing organisms in the soils after the application of sulphur. The finding is similar with that of (Classon and Ramaswami 1990). In general, sulfate mineralization as measured in a 0.15% calcium chloride extractant was found to be more closely related to CO_2 evolution. The higher correlation with CO_2 evolution for the calcium chloride and sodium acetate extractable sulphate then for the other extractants show that the 0.15% $CaCl_2$ should provide the better estimates of sulfate mineralized. Water soluble sulphur had a strong correlation with all the forms of sulphur (Borkotoki and Das 2008). At 30, 60 and 90th DOI. The highest 0.5M $NaHCO_3$ (Sodium bicarbonate) was observed in the treatment that received 30 mg S kg^{-1} (T_3) *i.e.* 49.20, 40.63 and 32.73 mg S kg^{-1} and it was on par with treatment receiving (T_4) 40 mg Skg^{-1}. Clear evidence was obtained for the presence of significant proportions of organic sulfate in soil extracts obtained with the bicarbonate extractants. No such indicators could be obtained for the 0.15% $CaCl_2$ extract investigated. The good extractability of available sulphur by 0.5M $NaHCO_3$ (pH 8.5) could be due to the fact that it extracts, in addition to the readily soluble sulphates, part of the adsorbed and organic sulphur. Similar finding by Kanwar and Mudahar (1986). The presence of carbon source is the main stimulant for the release of dehydrogenase enzyme. Dehydrogenase enzyme is responsible for organic matter oxidation in soil. The finding is similar with Ghani *et al.* (1990). Carbon –bonded S is comprised principally of amino acids such as methionine, cysteine, and sulpholipids (Neptune *et al*., 1975).

Table 2: Effect of different levels of sulphur on 500 ppm MCP(500 ppm Ca $(H_2PO_4)_2.H_2O$) extractant (ppm) at different days of incubation experiment.

Treatments	500 ppm Ca $(H_2PO_4)_2.H_2O$		
	30 DOI	**60 DOI**	**90 DOI**
T_0 - Control	21.36	28.89	31.99
T_1 - 10 mg sulphur /kg	29.15	35.54	40.30
T_2 - 20 mg sulphur/kg	38.09	44.50	47.59
T_3 - 30 mg sulphur/kg	51.76	60.27	67.34
T_4 - 40 mg sulphur/kg	45.80	51.67	56.41
SE(d) ±	1.83	1.99	2.12
$CD_{(p=0.05)}$	3.91	4.26	4.53

Table 3: Effect of different levels of sulphur on 0.5M NH_4OAc extractant (ppm) at different days of incubation experiment.

Treatments	0.5M NH_4OAc		
	30 DOI	**60 DOI**	**90 DOI**
T_0 - Control	19.11	22.53	24.79
T_1 - 10 mg sulphur /kg	24.27	29.73	34.04
T_2 - 20 mg sulphur/kg	29.24	33.36	36.31
T_3 - 30 mg sulphur/kg	39.23	44.28	49.03
T_4 - 40 mg sulphur/kg	29.86	37.80	42.06
SE(d) ±	1.21	2.42	2.83
$CD_{(p=0.05)}$	2.58	5.17	6.04

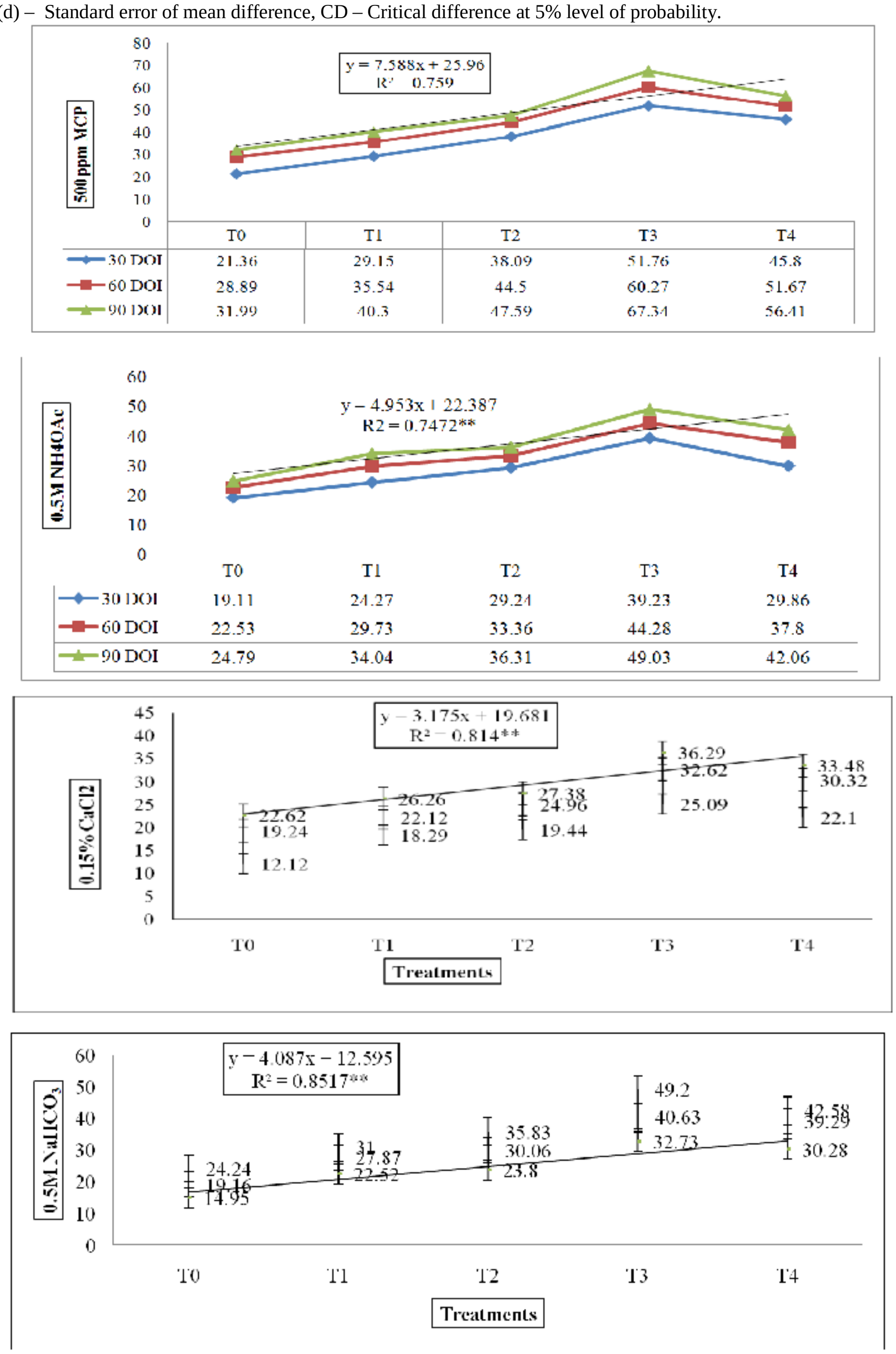
Table 4: Effect of different levels of sulphur on 0.15% $CaCl_2$ extractant (ppm) at different days of incubation experiment.

Treatments	0.15% $CaCl_2$		
	30 DOI	**60 DOI**	**90 DOI**
T_0 - Control	12.12	19.24	22.62
T_1 - 10 mg sulphur /kg	18.29	22.12	26.26
T_2 - 20 mg sulphur/kg	19.44	24.96	27.38
T_3 - 30 mg sulphur/kg	25.09	32.62	36.29
T_4 - 40 mg sulphur/kg	22.10	30.32	33.48
SE(d) ±	1.21	1.30	1.39
$CD_{(p=0.05)}$	2.58	2.77	2.96

Table 5: Effect of different levels of sulphur on 0.5M $NaHCO_3$ extractant at different days of incubation experiment.

Treatments	0.5 M $NaHCO_3$		
	30 DOI	60 DOI	90 DOI
T_0 - Control	24.24	19.16	14.95
T_1 - 10 mg sulphur /kg	31.00	27.87	22.52
T_2 - 20 mg sulphur/kg	35.83	30.06	23.80
T_3 - 30 mg sulphur/kg	49.20	40.63	32.73
T_4 - 40 mg sulphur/kg	42.58	39.29	30.28
SE(d) ±	1.60	1.37	1.24
$CD_{(p=0.05)}$	3.41	2.93	2.65

Notes:
$Ca(H_2PO_4)_2.H_2O$ = Monocalcium Phosphate (MCP), NH_4OAc = Ammonium acetate, 0.15% $CaCl_2$ = Calcium chloride, $NaHCO_3$ = Sodium bicarbonate, SE(d) – Standard error of mean difference, CD – Critical difference at 5% level of probability.



**Linear regression (r) is Significant at 1% (0.01) level; **Linear regression (r) is Significant at 5% (0.05) level.

Fig. 1. Linear regression relationship between treatments and extractants.

Table 6: Correlation co-efficients (r) of sulphur extractants during different days of incubation (DOI).

500 ppm $Ca(H_2PO_4)_2.H_2O$	**30 DOI**	**60 DOI**	**90 DOI**
30 DOI	1		
60 DOI	0.996443**	1	
90 DOI	0.99008**	0.997009**	1

0.5 M NH_4OAc	30 DOI	60 DOI	90 DOI
30 DOI	1		
60 DOI	0.980025**	1	
90 DOI	0.970251**	0.997483**	1

0.5M $NaHCO_3$	30 DOI	60 DOI	90 DOI
30 DOI	1		
60 DOI	0.976293**	1	
90 DOI	0.982621**	0.996797**	1

0.15% $CaCl_2$	30 DOI	60 DOI	90 DOI
30 DOI	1		
60 DOI	0.952033**	1	
90 DOI	0.955086**	0.993437**	1

Table 7: Correlation among extractants of sulphur during different days of incubation (DOI).

Extractants	**500 ppmMCP**	**0.5M NH_4OAc**	**0.5M $NaHCO_3$**	**0.15% $CaCl_2$**
500 ppmMCP	1			
0.5M NH_4OAc	0.988751**	1		
0.5M $NaHCO_3$	0.987212**	0.980279**	1	
0.15% $CaCl_2$	0.991108**	0.983748**	0.998325**	1

**Correlation co-efficients(r) is Significant at 1% (0.01) level

**Correlation co-efficients(r) is Significant at 5% (0.05) level

Sulphur transformation and its availability in soils depend on its various used of extractants. In order to judge the contribution of various extractants of sulphur towards the availability of sulphur in soil, it becomes imperative to work out the correlation within the different extractants of sulphur. The results of correlation among the different extractants of sulphur are presented in Table 6 and 7. The highest correlation of 500 ppm MCP was found between 60 DOI and 90 DOI (0.997009**), which was very closely followed by 30 DOI and 60 DOI for 0.5 M NH_4OAc (0.980025**), 0.5M $NaHCO_3$(0.996797**) at 60 DOI and 90 DOI, 0.15% $CaCl_2$ (0.993437**) at 60 and 90 DOI. The significant correlation between different extractants of S suggested an interrelated dynamic equilibrium among different extractants of Sulphur. The results revealed that the inter correlations between the extractable sulphur by different extractants were significantly correlated among themselves with varying degrees. The highest significant correlation was found between 0.5M $NaHCO_3$ and 0.15% $CaCl_2$ (0.998325**) followed by 500 ppm MCP and 0.15% $CaCl_2$ (0.991108**) and 500 ppm MCP and 0.5M NH_4OAc (0.988751**) gave least significant correlations with other extractants.

CONCLUSION

Results obtained in the present investigation, thus, revealed that distribution of different S extractants in surface layer of soils is greatly influenced by soil properties and inter-relationships amongst themselves. The release pattern of sulphur was highest up to 90 DOI except for sodium bicarbonate which was recorded at 30 DOI. Increasing levels of sulphur @ 30 mg S kg^{-1} increase the available sulphur content in the incubated soils. Soil texture (Clay content) was significantly influenced the release pattern of sulphur and higher the clay content, release of sulphur content also higher. Zero fertilization led to decline in the levels of all the extractants as compared to rest of the treatments. Gypsum treatment alone increased sulphur concentration in all four types of sulphur extractants. The results also indicated that different extractants of sulphur followed each other and are inter-related within them. The overall results suggested that S @ 30 mg kg^{-1} alone proved to be the best in respect of the parameters studied.

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Conflicts of Interest. None.

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