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Effect of Organic manures and Inorganic Sulphur on Releasing pattern, Adsorption and Desorption of Sulphur on Typic Haplustalf

P. Saravana Pandian *
Department of Soil and Environment,
Agricultural College and Research Institute,
Madurai- 625 104.

Laboratory experiments were carried out at the Agricultural College and Research Institute, Madurai during the year 2011 to evaluate the releasing pattern, adsorption and desorption behaviour of inorganic sulphur alone and in combination with organics in soil samples collected from farmer's field in Sivagangai district, Tamil Nadu. The results revealed that the rate of release of sulphur was increased upto 40 DAI and beyond which it got declined. At higher levels of sulphur, the amount of sulphur released was higher as compared to lower levels. Among the organics, the rate of release of sulphur was higher in vermicompost added treatment upto 50 DAI beyond which the amount of sulphur released was higher in press mud. Similarly, the adsorption of added sulphur was found to be higher in the treatment that has not received any one of the organic manures and the percentage of desorption of adsorbed sulphur was higher in the manurial treatments. The adsorption data were fitted well with the Langmuir adsorption isotherm as compared to the Freundlich isotherm.

Key words: Sulphur, Organics, Releasing Pattern, Sorption.

Sulphur has been recognised as the fourth major plant nutrient after N,P and K. Crops in general require as much sulphur as they need phosphorus. It is supplied to the soil from weathering of rocks and minerals, mineralized from the organic matter or from added fertilizer. A part of the released sulphate remain in soil solution and a part gets adsorbed or fixed on the soil colloidal complexes and its availability in the labile pool get reduced (Parfitt, 1982; Bhogal et al. 1996). The lability of sulphur in soil depends upon soil properties. Among these pH, presence of complexing anions (Lande et al, 1977), clay content and sesquioxides (Johnson and Todd, 1983; Fuller et al, 1985), $CaCO_3$ and native extractable sulphur are most important. Parfitt (1978) stated that due to ligand exchange mechanism, the organic anions make chelates with Fe and Al and reduces the adsorpti on of SO_4^{2-}. Evans (1986) reported that the SO_4^{2-} and the low molecular weight organic anions compete for the similar sorption sites. Based on the analysis of 1000 soil samples collected from the farmers holdings in Sivaganga district, more than 70 per cent of soil samples were found to be deficient in available S (< 10 mg kg-1). Therefore in order to know about the releasing pattern, adsorption and desorption behaviour of applied S alone and in conjoint with organic manures the present study was conducted

Materials and Methods

Bulk soil samples were collected from the experimental field from Sivagangai at 15 cm depth and processed. From this two hundred grams of soil was taken in plastic containers and imposed the treatments. The treatment structure consist of 5 levels of inorganic S (M_0 - Control, M_1 - 20 kg S ha^{-1}; M_2 - 40 kg S ha^{-1}; M_3 – 60 kg S ha^{-1}; M_4 – 80 kg S ha^{-1}) and 4 sources of organics (N_0-Control, N_1- press mud 5 t ha^{-1}; N_2 – FYM 5 t ha^{-1} and N_3 –vermicompost 5 t ha^{-1}). The experiment was conducted in a Factorial Completely Randomised Design with two replications. Sulphur was added in the form of gypsum and respective organic manure was added and incubated for 70 days at field capacity moisture level. Soil samples were drawn once in 10 days and analysed for available S using 0.15% $CaCl_2$ and the availability of S over a period of time was studied. Fifty grams of soil samples were taken in 250 ml polythene bottles to know the adsorption - desorption behaviour of S, Sulphur was added in the form of K_2SO_4 @ 0, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000 mg kg^{-1}. The same concentration of S were added to the following treatments also

- 50 g soil + Press mud @ 5 t ha-^{1}
- 50 g soil + FYM @5 t ha-^{1}
- 50 g soil + Vermicompost @5 t ha-^{1}

The contents were shaken in a reciprocating mechanical shaker for 24 hours (Barrow, 1970). The contents were centrifuged and the supernatant solution was decanted and S was estimated in an aliquot of the solution by turbidimetrically as per the procedure described by Chesnin and Yien (1951). From the amount of S lost from solution, the amount

*Corresponding author's email: pandian1968@ rediffmail.com

of S adsorbed by soil for each organic manure was calculated. The sorption data was fitted into the Langmuir and Freundlich adsorption equations.

Langmuir equation

$$\frac{c}{\frac{x}{m}} = \frac{1}{kb} + \frac{c}{b}$$

Freundlich equation

$$\log \frac{x}{m} \quad \log k \quad \frac{1}{n} \quad \log c$$

Where C = Equilibrium concentration of S in solution (mg l^{-1})

$\frac{x}{m}$ = Amount of S sorbed (mg kg^{-1})

b = S sorption maxima (mg kg^{-1})

k = Constant related to bonding energy

n and k = empirical constants.

After removing the supernatants, the soil samples in the bottles were washed five times with 50 ml portion of 0.5 N NH_4NO_3 and the washings were collected in 250 ml separate volumetric flasks. After the final washing, the volume was made upto 250 ml with 0.5 N NH_4NO_3 (Haque and Walmsley, 1972). The solution was analysed for SO_4^{2-} - S by turbidimetrically (Chesnin and Yien, 1951) and the amount of available S was calculated.

Results and Discussion

Effect of organic manures and incubation time on S availability

The results revealed that various organic manures significantly influenced the availability of S over a period of time (Table 1). It ranged from 12.8 to 17.4 mg kg^{-1} and the highest availability of 17.4 mg kg^{-1} was registered with the application of vermicompost 5 t ha^{-1} (N_3) closely followed by treatment receiving pressmud (N_1) however, they were statistically onpar with each other. Increased availability of S in vermicompost and pressmud might be due to narrow C:S ratio and active carbon pool present in vermicompost and pressmud. The incubation period also had a significant influence on the availability of S. It varied from 12.9 to 16.3 mg kg^{-1}. Significant increase in the available S was observed upto 40 DAI there after it got declined with advancement of incubation period. This might be due to formation of Fe-Al-SO_4 complex over a period of time which is relatively insoluble (Gowrisankar and Shukla, 1999).

The interaction effect of organic manures and incubation period had a significant influence on available S. In the case of pressmud (N_1), continuous linear increase was observed with the advancement

Table1. Effect of organic manures and incubation period on S availability (mgkg^{-1})

Organic sources	Incubation period (days)							Mean
	10	20	30	40	50	60	70	
N0	12.3	13.1	13.4	14.0	13.2	12.3	11.4	12.8
N1	13.1	14.2	15.8	17.6	18.4	19.0	19.4	16.8
N2	12.4	13.5	14.3	15.0	13.9	12.8	11.6	13.4
N3	13.7	15.6	17.2	18.4	18.6	18.7	18.3	17.2
Mean	12.9	14.1	15.2	16.3	16.0	15.7	15.2	

	SEd	CD (P=0.05)
N	1.3	2.7
I	0.8	1.7
NXI	1.4	2.9

Table 2. Effect of inorganic S and incubation period on S availability (mgkg^{-1})

Inorganic S	Incubation period (days)							Mean
	10	20	30	40	50	60	70	
M_0	7.9	9.2	9.6	10.7	10.7	10.3	10.3	9.8
M_1	10.7	12.5	13.6	15.7	15.6	15.2	14.3	11.7
M_2	13.3	14.7	15.8	16.8	16.6	16.3	16.1	15.7
M_3	15.4	16.4	17.5	18.1	18.1	17.7	17.2	17.2
M_4	17.2	17.2	19.5	20.1	19.5	19.0	18.3	18.7
Mean	12.9	14.0	15.2	16.3	16.1	15.7	15.2	

	SEd	CD (P=0.05)
N	1.3	2.7
I	1.0	2.1
NXI	1.4	2.9

of incubation period from 10 DAI to 70 DAI (13.1 to 19.4 mg kg^{-1}). While in the case of FYM (N_2) and vermicompost (N_3), a progressive increase in available S was registered upto 40 and 60 DAI respectively beyond which it got declined. The difference in releasing pattern of S among the organic manures may be due to the S content of the organic manures. The pressmud contain higher amount of S (1.2%) and this organic S could have been released slowly over a period to the labile pool.

Effect of inorganic S and incubation time on S availability

The rate of release of applied S was more at higher concentration as compared to the lower levels and it ranged from 9.8 to 18.7 mg kg-1 (Table 2). The highest available S content of 18.7 mg kg-1 was registered with the application of 80 kg S (M4) closely followed by 60 kg S ha-1 (M3) however, they were statistically on par with each other. At lower concentration, the rate of adsorption of sulphate over the soil colloidal complex would be higher to satisfy the positive charges exist on the soil colloidal complex (Sammi Reddy et al., 2001). As the experimental soil belonging to the sub group Typic Hyplustalf with 12.8 per cent sesquioxide, the applied S would have been adsorbed over sesquioxide and this could have been the possible reason for lower concentration of S at lower levels of S. The incubation time also had a significant influences on the availability of S and it ranged from 12.9 to 16.1 mg kg-1. A progressive increase in available S was noticed upto 40 days beyond which it got declined irrespective of levels of S. This might be due to continuous adsorption of sulphate over Fe- Al colloidal complex over a period of time as the experimental soil belong to the sub group Typic Haplustalf with 12.8 per cent sesquioxide content. Decrease in available S in labile pool over a period of time have already been confirmed by Athokpam et al. (2007). Similar to the individual effect, the interaction effect of levels of S and incubation time also had a significant influence on S availability. The availability of S followed a quadratic relationship over a period of time irrespective of levels of S.

Effect of organic manures and inorganic S on availability of S

The data depicted in Table 3 revealed that both organic manures and inorganic S had a significant effect on release of S at different days of incubation. The rate of release of applied S was more at higher concentration as compared to the lower levels and it ranged from 9.8 to 18.7 mg kg-1. The highest available S content of 18.7 mg kg-1 was registered with the application of 80 kg S ha-1. Since at lower concentration, the rate of adsorption of sulphate over the sesquioxide would be higher to satisfy the unsatisfied positive charges exist on the soil colloidal complex (Tiwari and Gupta, 2006). In the case of organic manures, the available S content varied from 12.8 to 17.2 mg kg-1 and the higher available content of 17.2 mgkg-1 was registered with the application of vermicompost (N3) closely followed by pressmud (16.9 mg kg-1).Interaction effect of organic manures and inorganic S was prominent on the release of S irrespective of the days of incubation. The double combination of vermicompost with 80 kg S ha-1 registered the highest available S content (21.3 mg kg-1) closely followed by pressmud with 80 kg S ha-1. This might be due to higher S content of vermicompost (1.1%) and pressmud (1.2%) which would have favoured the long term release of S into the labile pool. The positive relationship between S content, Arylsulphatase activity and release of S was reported by Shriner and Henderson (1978).

Adsorption of sulphur

The data on S adsorption revealed that amount of S adsorbed follows the order as Control (N_0) > FYM (N_1) press mud (N_2) > vermicompost (N_3). An increase in S adsorption was noticed upto 16.0, 18.0 and 16.0 mg l^{-1} for press mud, FYM and vermicompost treatments respectively beyond which it got declined

Table 3. Effect of organic manures and inorganic S on S availability (mgkg-1)

inorganic S	Organic manures				Mean
	N0	N1	N2	N3	
M_0	6.1	12.0	8.6	12.4	9.8
M_1	12.3	15.5	12.3	15.9	14.0
M_2	14.0	17.4	13.9	17.5	15.7
M_3	15.5	19.1	15.2	19.1	17.2
M_4	16.3	20.5	16.7	21.3	18.7
Mean	12.8	16.9	13.3	17.2	

	SEd	CD (P=0.05)
M	1.6	3.3
N	1.3	2.7
MXN	1.7	3.5

(Table 2). Soil belonging to the manured treatments possessed lower S adsorption capacity (55%) than the unmanured one (71%). However, among the organic manures the lowest S adsorption was registered in vermicompost (50.6%) followed by press mud (52.9%) and FYM (61.7%). This may be attributed to the narrow C:S ratio (41:1) and active carbon pool present in vermicompost. The lowest amount of adsorption of S in manured soils may be attributed to the competition between the organic

Table 4. Effect of organic manures and inorganic S on adsorption and desorption of S in soils

S added ($mgkg^{-1}$)	Initial S concentration ($mg1^{-1}$)	Equilibrium S concentration ($mg1^{-1}$) (C)	Amount of S adsorbed (mg kg^{-1}) (x/m)	c/x/m	% of added S adsorbed	Amount of S desorbed (mg kg^{-1})	% of S desorbed
			N_0 (Control)				
0	-	-	-	-	-	6.4	-
100	2	0.25	83.0	0.003	83	18.5	22.3
200	4	0.50	165.5	0.003	82.8	27.4	16.5
300	6	1.18	235.0	0.005	78.3	32.5	13.8
400	8	1.65	300.0	0.006	75.0	40.5	13.5
500	10	2.65	358.5	0.007	71.7	47.4	13.2
600	12	3.50	412.0	0.008	68.7	54.5	13.2
700	14	4.45	450.0	0.010	64.3	60.0	13.3
800	16	5.30	515.5	0.010	64.4	74.8	14.5
900	18	6.00	558.0	0.011	62.0	90.0	16.1
1000	20	6.70	600.0	0.011	60.0	115.5	19.3
			N_1(Pressmud)				
0	-	-	-	-	-	10.0	-
100	2	0.60	58.5	0.010	58.5	27.0	46.2
200	4	1.30	118.0	0.011	59.0	36.0	30.5
300	6	2.45	172.5	0.014	57.5	45.5	26.4
400	8	3.30	228.5	0.014	57.1	54.5	23.9
500	10	4.20	275.5	0.015	55.1	59.5	21.6
600	12	5.20	340.0	0.015	56.7	67.0	19.7
700	14	6.30	382.5	0.016	54.6	78.5	20.5
800	16	7.95	415.5	0.019	51.9	92.5	22.3
900	18	9.70	385.0	0.025	42.8	107.5	27.9
1000	20	11.2	360.0	0.031	36.0	125.0	34.7
			N_2 (FYM)				
0	-	-	-	-	-	8.00	-
100	2	0.40	72.50	0.005	72.5	24.5	33.8
200	4	0.75	140.5	0.005	70.3	30.0	21.4
300	6	1.45	205.0	0.007	68.3	38.5	18.8
400	8	2.25	265.0	0.008	66.3	46.0	17.4
500	10	3.10	313.5	0.010	62.7	54.5	17.4
600	12	3.95	360.0	0.011	60.0	63.5	17.6
700	14	4.85	408.5	0.012	58.3	74.0	18.1
800	16	5.90	445.0	0.013	55.6	86.5	19.4
900	18	7.50	500.0	0.015	55.6	100.0	20.0
1000	20	8.25	475.0	0.017	47.5	121.5	25.6
			N_3 Vermi compost				
0	-	-	-	-	-	10.5	-
100	2	0.75	55.00	0.014	55.0	28.5	51.8
200	4	1.35	114.5	0.012	57.3	36. 5	31.9
300	6	2.60	160.0	0.016	53.3	46.5	29.1
400	8	3.45	215.0	0.016	53.7	55.0	25.6
500	10	4..35	265.5	0.016	53.1	61.5	23.2
600	12	5.20	328.5	0.016	54.8	69.0	21.0
700	14	6.35	365.5	0.017	52.2	81.5	22.3
800	16	8.00	400.0	0.020	50.0	94.5	23.6
900	18	9.85	375.5	0.026	41.7	110.0	29.3
1000	20	11.30	345.0	0.033	34.5	128.0	37.1

anions and SO_4^{2-} for the same adsorption sites. Evans (1986) reported that the SO_4^{2-} and the low molecular weight organic anions compete for the similar adsorption sites.

The unmanured soil recorded higher percentage of adsorption of added S. This may be attributed to the specific adsorption of SO_4^{2-} by the hydrous oxides of Fe and Al (Singh, 1984). The experimental soil is belonging to the sub group Typic Haplustalf with 12.8 per cent sesquioxide. Due to high sesquioxide, more amount of SO_4^{2-} would have been retained in the unmanured soil. Neary *et al.* (1987) reported that the amount of inorganic fractions of soil matrix, amorphous and crystalline forms of Fe and Al found to control the SO_4^{2-} retention kinetics of podzols. A significant relationship between the SO_4^{2-} adsorption and Fe_2O_3 (r= 0.820**) and Al_2O_3 (r=0.859**) was reported by Dolui and Nandi (1987). The result showed that adsorption of S increased with the increasing concentration of added S upto 800 mg kg^{-1} beyond which it got declined except in unmanured soil (N_0). The percentage of adsorption of S was found to be higher at the lower concentration of S (100 mg kg^{-1}) and decreased gradually with the corresponding increase in the concentration. These results are in line with the earlier works of Bhogal *et al*. (1996) who found that the adsorption of SO_4^{2-} decreased with the increasing concentration of added S in Calciorthents.

Adsorption isotherm

The adsorption data were fitted to two adsorption isotherms viz., Langmuir and Freundlich equations. The results revealed that the S adsorption was fitted well in the Langmuir adsorption isotherm by registering higher R^2 values (0.96**) than the Freundlich isotherm (R^2= 0.94**). Fox (1982) compared four adsorption equations to explain the SO_4^{2-} adsorption and the highest regression coefficient was noticed with Langmuir equations. Similarly Sammi Reddy *et al.* (2001) and Saravana Pandian and Saravanan (2012) reported that the Langmuir equation gave the best fit over Freundlich equations. As regard the Langmuir adsorption equation, the SO_4^{2-} sorption maxima (b) ranged from 484 mg kg^{-1} in vermicompost to 654 mg kg^{-1} in unmanured control treatments respectively. The highest and the lowest SO_4^{2-} sorption maxima were registered in control and vermicompost treatments respectively. Among the manurial treatments, the highest SO_4^{2-} adsorption maxima was recorded in FYM (N_2) followed by press mud (N_1) and vermicompost (N_3) treatments. Similar to the adsorption maxima, the bonding energy (k) values were extended from 0.288 to 0.357. The highest k value was noticed in unmanured control (N_0) (0.357) followed by FYM (N_2) treatments. This may be due to the high sesquioxide content, the SO_4^{2-} would have been adsorbed strongly over the Fe and Al oxides to satisfy their positive charges. Johnson and Todd (1983) and Stankogolden *et al*. (1994) observed a positive relationship between the SO_4^{2-} adsorption maxima and the sesquioxide in laterite soils. The lower values of adsorption maxima and bonding energy coefficient in manure added soil may be attributed to the saturation of adsorption sites by organic anions (Kodama and Schnitzer, 1980).

In the case of Freundlich adsorption isotherm, the constants viz, K and 1/n values varied from 2.35 to 2.50 and 0.329 and 0.603 respectively. The highest and the lowest K values were registered with unmanured control (N_0) and vermicompost (N_3) treatments respectively. In general, the K values were

Table 5. Langmuir and Freundlich constants of S adsorption in soil as influenced by organic manures and inorganic S.

(A) Langmuir equation

Treatments	Langmuir constants		Regression equation	R2 (n=10)
	SO4 sorption maximum (b) (mg kg-1)	Bonding energy (K)		
N0	654	0.357	Y=0.0014+ 0.0061X	0.99**
N1	510	0.315	Y= 0.0026+0.0037X	0.96**
N2	560	0.340	Y= 0.0021+0.0063X	0.96**
N3	484	0.288	Y= 0.0035+ 0.0128X	0.92**

(B) Freundlich equation

Treatments	Langmuir constants		Regression equation	R2 (n=10)
	K	1/n		
N0	2.50	0.329	Y=0.90+0.329X	0.96**
N1	2.37	0.431	Y= 0.85+0.431X	0.93**
N2	2.44	0.431	Y= 0.88+0.431X	0.96**
N3	2.35	0.603	Y= 2.35+ 0.603X	0.91**

higher in unmanured treatments than the manured ones. As regards the manurial treatments, the highest K value of 2.50 was recorded in N_0 followed by 2.44 in N_2 treatments. The concentration exponent (1/n) values were higher in the treatments received organic manures as compared to the unmanured control and the highest and lowest values of 0.603 and 0.329 were recorded in vermicompost and unmanured control respectively.

Desorption of sulphur

The data on desorption of added S indicated that it extended from 18.5 to 115.5, 27.0 to 125.0, 24.5 to 121.5 and 28.5 to 128.0 mg kg^{-1} in control (N_0), press mud (N_1) FYM (N_2) and vermicompost (N_3) treatments respectively. The results showed that the lowest amount of desorption was registered in unmanured control (N_0) whereas, the highest quantity was recorded in N_3 (vermicompost). It was observed that the amount of desorption was higher in the manured soils which may be due to presence of SO_4^{2-} in an adsorbed from over organic colloidal constituents which would be released at a faster rate upon equilibrium with 0.15% $CaCl_2$ $2H_2O$. Besides, due to the high content of organic colloids in the manure added soils, the organic anions would have chelated the metallic cations like Fe and Al which inturn released the SO_4^{2-} to the labile pool. Evans and Anderson (1990) observed a positive relationship between the organic matter and desorption of SO_4^{2}. Among the manured soils, the highest rate of desorption was registered in vermicompost added soils than the others. This may be attributed to narrow C: S ratio (41; 1) of vermicompost.

Conclusion

From this study, it is concluded that the SO_4^{2-}-S get adsorbed strongly over sesquioxides in Typic Haplustalf To supply the SO_4^{2-} continuously for the crop growth and to reduce the adsorption of SO_4^{2-}, application of organic manures along with sulphur fertilizer has become very much essential. Among the organic manures, application of vermicompost (or) press mud has improved the availability of SO_4^{2-} in labile pool.

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