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Sorption and desorption behavior of sulphur in soils of Tamil Nadu, India

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Abstract
Adsorption and desorption behaviour of S in soil, the soils were collected at 15 cm depth from the four major soil series (Peelamedu, Palaviduthi, Vylogam and Irugur) of pulses growing areas of Madurai district. From the adsorption study, it was found that the maximum adsorption of sulphur was registered in Palaviduthi series followed by Irugur, Vylogam and Peelamedu series. The adsorption data were fitted in two adsorption isotherms viz., Langmuir and Freundlich equations. Among the two adsorption isotherms, Langmuir isotherm fitted well for sulphur adsorption. With respect to Langmuir adsorption isotherm, the SO_4^{2-} sorption maxima (b) was observed in the order of Peelamedu > Vylogam > Irugur > Palaviduthi series. The bonding energy was recorded in the order of Irugur > Palaviduthi > Vylogam > Peelamedu series. The Maximum buffering capacity observed in the order of Peelamedu > Palaviduthi > Vylogam > Irugur series. The sorption and desorption capacity of these soils varied moderately with the variations in pH, iron and aluminum oxides, available phosphorus, exchangeable calcium plus magnesium, clay content and organic carbon content of the experimental soils. The data, thus obtained were employed to work out maximum buffering capacity (b) of sulphate sorption, the high value of which essentially favored the sorption processes in these soils. Soils having greater irreversible sulphate sorption capacity require adequate sulphur in order to ensure optimum pulses growth and yield.

Keywords: sulphate adsorption and desorption, sorption and desorption isotherm, Pulses.

1. Introduction

Sulphur represents the ninth and least abundant essential macronutrient in plants, preceded by C, O, H, N, K, Ca, Mg and P. The dry matter of sulphur in plants is only about one-fifteen of that of nitrogen. Sulphur plays various critical roles in catalytic or electrochemical function of biomolecules in the cells (Saito, 2004) [26]. Among the macronutrient N, K, Ca are non toxic to an extent at low to moderate concentrations while P and S seems to be more or less toxic to plants at higher concentration (Goldbold and Huttermann, 1985; Niess, 1999) [14, 21]. Even the green revolution has not increased the yield of pulses, indeed its emphasis on cereals which has often led to decrease in legume production (Bhatnagar, 1985). The reasons for decreasing pulse productivity in India are cultivation of pulses in marginal and poor soil conditions, cultivation under rainfed situation, cultivated as rice fallow, imbalanced nutrient management practices, improper irrigation management, minimum care by the farmers and grown in off-season. Modern intensive farming has resulted in higher demand for fertilisers because of removal of all the essential nutrients in higher proportions by the crops. Most of our attention for fertiliser use has been restricted to the use of N, P and K, the three primary nutrients required by the crops in large quantities. Sulphur nutrition to crops has not been fully realised during the past mainly because of the fact that S deficiency was not a serious problem. Recently interest on S as a plant nutrient has increased dramatically. It general crops require as much as S as they need P. It is indispensable for the synthesis of certain amino acids like cysteine, cystyine and methionine besides being involved in various metabolic and enzymatic processes of plants. It is also a constituent of protein and glutathione, a compound that play a part in plant respiration and synthesis of essential oils.

For managing sulpur, sulphate sorption and desorption is important. The concentration of sulphate in soil solution, as predicted by the sorption and desorption isotherms, provided valuable information on sulphur availability to crop plants (Reddy *et al*. 2001; Kimsey *et al*. 2005) [25, 18]. The study area have charnockites and khondalites of Archaean age are the two main rock types are encountered. Charnockites include acid charnockite and related migmatites with bands of basic granulite and magnetite and quartzite.

The khondalite group of rocks consists of crystalline limestone, calc – gnesis, calc – granulite, garnet sillimanite gnesis, hornblende and biotite gnesis and related migmatites with bands of quartzite. They are exposed mainly with acid intrusive of pink and gneissic granite. Minor pegmatite and quartz veins are seen throughout. Indeed most of the study area are deficient in S due the calcic and acidic nature. Sulphur deficiencies have been reported in crops such as groundnut, clover, cotton, alfalfa, tobacco, maize, wheat, rice, tea, citrus and cruciferous crops (Tandon, 1991) [30]. Several workers have attempted in various soils to assess the S status and its availability to the plants. Consolidation of information shows that S deficiency in Indian soils is more extensive than in generally thought. The sulphur status of major pulses growing soils of Madurai district is lacking. Besides the information regarding, correct source and levels of sulphur recommended for blackgram is also not available. Therefore, inorder to shed some light on the thrust areas, the study has been contemplated to evaluate the sources and levels of sulphur for maximizing the productivity of blackgram in Madurai district, Tamil Nadu. However, earlier research on sulphate sorption and desorption in different soils revealed that several factors might be responsible for sulphur deficiency; these include pH (Arbestain *et al*. 1999) [2], the presence of complexing anions (Parfitt 1982) [22], extractable aluminium and iron (Johnson and Todd 1983; Dolui and Jana 1997; Reddy *et al*. 2001) [17, 9, 25], organic carbon (Gerstl and Mingelgrin 1984; Evans 1986) [13, 11], calcium carbonate content (Bhogal *et al*. 1996) [3] and native extractable sulphate (Shanley 1992) [28].

2. Materials and methods
Brief description of study area
Madurai district, to stay at rest on the Southern part of the Tamil Nadu is bounded by the districts of Sivagangai on the East, Theni on the West, Virudhunagar on the South and Dindugal on the North. It is situated in between 9°30' and 10°30' north latitudes 77°30' and 78°30' east longitudes. The total geographical area of the district is 3741.73 sq km and divided in to 13 blocks. Out of 13 blocks in Madurai district, six blocks were selected for delineation of sulphur status on pulses growing areas such as Usilampatti (9.97 N° - latitude, 77.8 E° - longitude), T. Kallupatti (9.93N° - latitude, 78.12° - longitude), Thiruparankundram (latitude – 9.89 N°, longitude- 78.07 E°), Thirumangalam (latitude - 9.91 N°, longitude - 77.98 E°), Kallikudi (latitude - 9.65N°, longitude - 77.96 E°) and Chellampatty (latitude - 9.97 N°, longitude - 77.69 E°) with the altitude of 101 m above mean sea level. Red loam and black soils are the major soils in Madurai district. Vaigai River is the main source of irrigation in this district.

Basic properties of the experimental site
The results of the four soil series such as Peelamedu, Palaviduthi, Vylogam and Irugur were in pH of 8.52, 7.21, 7.10 and 7.19 and electrical conductivity of 0.60, 0.45, 0.31 and 0.19 dSm^{-1} respectively (Table 1). The exchangeable Ca status of the soils was 12.2, 8.30, 7.30 and 5.30 c mol (p+) kg^{-1} and the exchangeable Mg status of the soils was 5.76, 3.52, 2.32 and 2.12 c mol (p+) kg^{-1}. The CaO and MgO per cent of the soils were 17.1, 11.6, 10.2 and 7.42 and 9.61, 5.87, 3.87 and 3.54 per cent in four soil series respectively. The total sesquioxides (R$_2$O$_3$) content of the soils were 4.60, 12.6, 12.8 and 12.7 per cent. The cation exchange capacity of the soils was 26.2, 17.2, 17.5 and 8.13 c mol (p+) kg^{-1} and the anion exchange capacity was 2.01, 5.09, 5.30 and 5.12 c mol $_c$ kg^{-1}.

The organic carbon status of the soils was 5.20, 3.60, 4.80 and 2.40 g kg^{-1}.

Adsorption and Desorption behaviour of sulphur
To study the adsorption and desorption behaviour of S in soil, the soils were collected at 15 cm depth from the four major soil series (Peelamedu, Palaviduthi, Vylogam and Irugur) of pulses growing areas of Madurai district. The soils were dried in shade and sieved through 0.5 mm sieve and stored in polythene bags. Ten gram of soils from four soil series was taken in 250 ml polythene bottle separately. Sulphur was added @ 50 ml in the form of K$_2$SO$_4$ at the rate of 0, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000, 1100, 1200, 1300, 1400, 1500 and 1600 ppm of S. 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) [6]. From the amount of S lost from the solution, the amount of S adsorbed by soil at each level of S was calculated.
The sorption data was fitted in to the Langmuir and Freundlich equations.

> *Langmuir equation*: $C/x/m = 1/Kb + C/b$
> *Freundlich equation*: $\log x/m = \log k + (1/n) \log C$

Where,
C = Equilibrium concentration of S in soil solution ($\mu g \ ml^{-1}$)
x/m = Amount of S sorbed ($\mu g \ g^{-1}$)
b = S sorption maxima ($\mu g \ g^{-1}$)
K = constant related to bonding energy
n and k = empirical constants

After shaking, the supernatant was separated by centrifuging and the soil samples in the separating bottle were washed five times with 50 ml portion of 0.5 N NH$_4$NO$_3$ and the washings were collected in a volumetric flask. After final washing, the volume was made upto 250 ml with 0.5 N NH$_4$NO$_3$ (Hauqe and Walmsley, 1973). The solution was analysed for SO$_4^{2-}$ - S by turbidimetrically (Chesnin and Yien, 1951) [6] and the amount of SO$_4^{2-}$ was calculated.

3. Results and Discussion
Vylogam series (Typic Rhodustalf)
The data on S adsorption revealed that the amount of adsorbed S was increased with the increase in the concentration of S up to 900 $\mu g \ ml^{-1}$ beyond which it got declined (Table 1). The percentage of S adsorption was ranged from 11.2 to 47.9 per cent. The higher percentage of adsorption S was recorded upto the concentration of 500 $\mu g \ ml^{-1}$ beyond that it got declined gradually. The SO$_4^{2-}$ adsorption can be either specific or non-specific, in the non-specific adsorption the electrostatic (coloumbic) forces are involved and the specific adsorption occurs by ligand exchange. This series soil had higher amount of sesquioxides (12.8%) so the non-specific adsorption could have occurred with high levels of free iron and aluminium oxides and hydroxides. So the hydroxyl aluminium bonded polymer (Fe-Al- SO$_4^{2-}$) or (Fe- SO$_4^{2-}$- O-H- Fe) or (Al- SO$_4^{2-}$- O-H-Al) clays would have been formed and broken edges of the clays might have protonated surface for non-specific adsorption, so the adsorption SO$_4^{2-}$would have been higher. This result are in confirmation with the findings of Rajan (1978) [24] and Fuller *et al*. (1985) [12] who illustrated that the complete reversibility

would be expected only if all the adsorbed SO_4^{2-} would have been non-specifically adsorbed.

The adsorption data were fitted in two adsorption isotherms viz., Langmuir and Freundlich equations (Table 6). With respect to Langmuir adsorption isotherm, the SO_4^{2-} sorption maxima (b) was observed of 454.54 (mg kg $^{-1}$), bonding energy (K) 0.51 and maximum buffering capacity was 231. 81 (mg kg $^{-1}$) and the highest relationship was observed in Langmuir equation ($R^2 = 0.82**$) which is highly correlated to equilibrium S concentration (C) and S adsorption (x/m).

In the case of Freundlich adsorption isotherm, the constants viz., k and 1/n values have exhibited the same trend as that of Langmuir isotherm. The highest concentration exponent (1/n = 1.007), K (3.516) and the regression coefficient (R = 0.53**) were registered in Vylogam series. This might be due to higher maximum buffering capacity (231. 81 mg kg $^{-1}$) of soil suggest a concomitant greater degree of resistance in concentration of sulphur in soil solution and continuous application of S might have increased the residual S in the soil by the fate of fixation or adsorption to satisfy the positive surface. This result was in line with the findings of Saravana Pandian (2010) [27] who revealed that the S adsorption was fitted well in the Langmuir isotherm registering higher R^2 value ($R^2 = 0.945**$) than the Freundlich isotherm ($R^2 = 0.670**$).

 In Vylogam series the amount of S desorbed was recorded as 165 μg $^{-1}$ while adding S @ 1000 μg ml^{-1} beyond it got declined. The percentage of S desorption was ranged from 27.0 to 52.5. The rate of desorption of S was increased linearly while adding S from 0 to 1600 μg ml^{-1}. Because of high tenacity this soils might have adsorbed more sulphate and desorbed lesser. The positive reason may be due to adsorption of anion on positive surface of the iron and aluminium oxides and later it would have greater non-specific sorption by displaced hydroxyl ions produces metal ion pairs (Fe-O-S-O-Fe) or (Al-O-S-O-Al) complexes. Indeed at higher concentration, the rate of desorption was increased upto 1000 μg ml^{-1} of added S after that the reversibility of S was taken place. Similar observations were made by Comfort *et al.* (1992) [7] and Alves and Lavorenti (2006) [1].

Palaviduthi series (Typic Haplustalf)
The adsorption of S increases with the increase in the concentration of S up to 800 μg ml^{-1} beyond which it got declined in Typic Haplustalf (Table 3). The percentage of adsorption was ranged from 13.5 to 53.4 per cent. The highest percentage of adsorption S was registered in 600 μg ml^{-1} beyond that it got decreased gradually. This might be attributed to the higher sesquioxides (12.6%) and low exchangeable Ca (8.30 c mol (p$^+$) kg^{-1}) content of soil would have adsorbed higher amount of sulphate through electrostatistically as counter ion in the positive surface of diffuse double layer to satisfy the positive charges and less reversibility has taken place. Similar finding was reported by Hingston *et al.* (1968) [16]. According to Dolui and Nandi (1989) [10] who reported that a very close and significant correlation of adsorbed sulphate with free Fe_2O_3 (r= 0.82**) and Al_2O_3 (r= 0.859**) was found and also indicated that the adsorption of sulphate sulphur depends upon the content of free Fe_2O_3 and Al_2O_3.

The adsorption data were fitted into two adsorption isotherms viz., Langmuir and Freundlich equations. In Langmuir adsorption isotherms, the SO_4^{2-} sorption maxima (b) was 769.23 mg kg $^{-1}$, bonding energy was (K) 0.33, Maximum buffering capacity was 253. 84 mg kg $^{-1}$ and the highest

relationship was observed in Langmuir equation ($R^2 = 0.90**$) which it is highly correlated with equilibrium S concentration (C) and S adsorption (x/m). In Freundlich adsorption isotherm, the constants viz., k and 1/n values registered the same trend as that of Langmuir equation. The concentration exponent (1/n) values was 0.924, K was 4.118 and R^2 0.46**. The highest positive relationship with equilibrium S concentration (C) and S adsorption (x/m) was exhibited in Langmuir as compared to Frendlich isotherm. This has been attributed to the high ZPC (zero point charge) and presence of specific surface which would have given high tenacity to hold the sulphate ions in their colloidal complex. This result was in concurrence with the earlier findings of Parks (1965) [23] and Cornell and Schwertmann (1996) [8].

The S desorption was ranged from 6 to 133 μg g $^{-1}$ and the highest desorption of 133 μg g $^{-1}$ at 1300 μg ml^{-1} beyond which it got declined was recorded. The percentage of S desorbed ranged from 23.5 to 51.1. The rate of desorption was increased while adding S upto 1300 μg ml^{-1} beyond which it got decreased. This might be due to the high tenacity to bind the sulphate on sesquioxide complexes, which is evident from the lesser desorption of sulphate with high bonding energy (K of 4.118 in Frendlich and Langmuir K = 0.33). This finding was in agreement with McDonald and Hart (1990) [19] who reported that an increase in the rate of adsorption of S in the soils with higher ambient S concentration.

Peelamedu series (Typic Haplustert)
The amount of S adsorption increased with the increase in the concentration of S upto 900 μg ml^{-1} beyond which it got declined (Table 4 & 6). Regarding added S, the percentage of adsorption was ranged from 15.2 to 52.1. The higher percentage of adsorption S was registered in the lower concentration and later it got declined gradually. This might be due to the soil which had high amount of exchangeable Ca(12.2 c mol (p+) kg^{-1}), high exchangeable Mg (5.76 c mol (p+) kg^{-1}) and high CaO (17.1%) which would have caused higher sorption by coadsorption of Ca^{2+} and SO_4^{2-} as a $CaSO_4^{2-}$ ion pair complex. This result was in line with the findings of Bolan *et al.* (1993) [5] who illustrated that at higher Ca^{2+} concentration (0.003–0.015 mol L^{-1}) the adsorption of SO_4^{2-} was increased.

The adsorption data were fitted into two adsorption isotherms viz., Langmuir and Freundlich equations. In Langmuir adsorption isotherm, the SO_4^{2-} sorption maxima (b) was observed of 172.41 mg kg $^{-1}$, bonding energy was (K) 1.87 and maximum buffering capacity was 322.40 mg kg $^{-1}$. The highest relationship was observed in Langmuir equation ($R^2 = 0.84**$) which was highly correlated to equilibrium S concentration (C) and S adsorption (x/m). In Freundlich adsorption isotherm, the constants k and 1/n values were recorded as that of Langmuir equation. The concentration exponent (1/n) value was 1.06, K was 3.692 and R^2 0.54**. The highest positive relationship with equilibrium S concentration (C) and S adsorption (x/m) was exhibited in Langmuir when compared to Frendlich isotherms. This might be attributed to the high bonding energy (K= 1.87 in Langmuir) and Maximum buffering capacity (322.40 mg kg $^{-1}$) when compared to other series which implies high affinity for sorption increases exponentially with decreasing saturation in surface. This may lead to induce high retention of sulphate in solution phase so desorption was higher in Peelamedu series. This result was in concurrence with the findings of Das *et al.* (2009) who reported that maximum buffering capacity of soil is a fundamental characteristic to

sulphate release or retention property by integrating the intensive and extensive components of adsorption.

With respect to S desorption, it recorded 174 µg g^{-1} at 900 µg ml^{-1} concentration of added S beyond it got declined. The percentage of S desorbed was ranged from 23.4 to 47.7. The rate of desorption of S was significantly increased while adding S upto 1600 µg ml^{-1}. This high desorption might be due to low sorption maxima and high buffering capacity through hysteresis effect and phosphate ions in the soil also compete the sorption sites which leads to attain higher desorption of S. This result was agreement with the findings of Saravana Pandian (2010) [27] who found that the SO_4^{2-} adsorbed on the surface of the soil colloids would be released easily to the solution phase and the chemisorbed SO_4^{2-} would be moved slowly to the exchangeable site to maintain the equilibrium which in turn would be released in to the labile pool.

Irugur series (Typic Ustropept)
The data on S sorption revealed that the amount of S adsorbed increased with the increase in the concentration of S upto 800 µg ml^{-1} of added S beyond which it got declined (Table 5 & 6). Regarding added S, the percentage of adsorption was ranged from 8.1 to 49.0 per cent. The higher percentage of adsorption S was registered in the lower concentration and it got declined gradually. This may be due to the higher free iron and aluminium oxides (12.7%) found in the soils which sorbed reversible sulphate ions in their positive charged surfaces. This was supported by Singh (1984) [29] who attributed to the specific adsorption of SO_4 by the hydrous oxides of Fe and Al.

The adsorption data were fitted into two adsorption isotherms viz., Langmuir and Freundlich equations. Regarding Langmuir adsorption isotherm, the SO_4^{2-} sorption maxima (b) was observed at 714.28 mg kg^{-1}, bonding energy was (K) 0.21 and maximum buffering capacity was 149.99 (mg kg^{-1}). The highest relationship was observed in Langmuir equation (R^2 = 0.93**) which was highly correlated to equilibrium S concentration (C) and S adsorption (x/m). In Freundlich adsorption isotherm, the constants viz., k and 1/n values were recorded as that of Langmuir equation. The concentration exponent (1/n) values was 0.796, K was 3.5837 and R^2 was 0.46**. The highest positive relationship with equilibrium S concentration (C) and S adsorption (x/m) was exhibited that the amount of adsorbed S had a positive relationship and also highly significant in Langmuir when compared to Freundlich isotherm. This might be attributed to high sorption by positive surface sesquioxide form of Fe-SO_4 or Al-SO_4 complexes. This was supported by Goutam Kumar Ghosh and Nishi Ranjan Dash (2012) [15].

The maximum S desorption of 116 µg g^{-1} was registered in 1000 µg ml^{-1} concentration of added S beyond it got declined. The percentage of S desorption ranged from 20.4 to 57.6 and it was significantly increased while adding S upto 1600 µg ml^{-1}. Regarding this series, the desorption was lower when compared to other soil series because in this series the adsorption was occurred by non-specific by the sulphate ions which are held electrostatically as a counter ion in the electric double layer next to positive surface soil colloids rich in free sequioxides. This result was in line with the findings of Marsh *et al*. (1987) [20]. According to Goutam Kumar Ghosh and Nishi Ranjan Dash (2012) [15] who reported that amount of sulphate desorbed by KH_2PO_4 varied considerably among the soils and the desorbed amounts increased in proportion to amounts of sulphate sorbed.

Table 1: Basic properties of four major soil series of pulses growing areas in Tamil Nadu

Parameters	Peelamedu (Typic Haplustert)	Palaviduthi (Typic Haplustalf)	Vylogam (Typic Rhodsutalf)	Irugur (Typic Ustropept)
pH	8.52	7.21	7.10	7.19
EC (dSm^{-1})	0.60	0.45	0.31	0.19
Exchangeable Ca(c mol (p+) kg^{-1})	12.2	8.30	7.30	5.30
Exchangeable Mg(c mol (p+) kg^{-1})	5.76	3.52	2.32	2.12
CaO (%)	17.1	11.6	10.2	7.42
MgO (%)	9.61	5.87	3.87	3.54
R_2O_3 (%)	4.60	12.6	12.8	12.7
CEC (c mol (p+) kg^{-1})	26.2	17.24	17.5	8.13
AEC (c mol$_c$ kg^{-1})	2.01	5.09	5.30	5.12
OC (g kg^{-1})	5.20	3.60	4.80	2.40

Table 2: Langmuir and Freundlich Constant for S adsorption in pulses growing areas in Madurai District
A). Langmuir Constants

Series	Langmuir constants		Maximum buffering capacity	Regression equation	R^2
	SO_4^{2-} sorption maximum (mg kg^{-1})	Bonding energy (K)			
Palaviduthi	769.23	0.33	253.84	0.0039x+ 0.0013	0.90
Peelamedu	172.41	1.87	322.40	0.0031x+ 0.0058	0.84
Irugur	714.28	0.21	149.99	0.0065x + 0.0014	0.93
Vylogam	454.54	0.51	231.81	0.0043x + 0.0022	0.82

B). Frendlich Constants

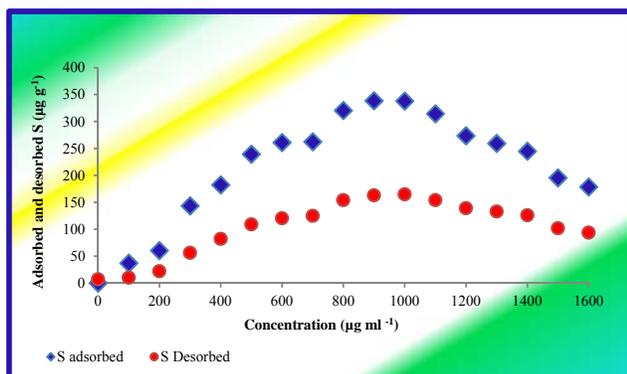| Series	Frendlich constants		Regression equation	R^2
	K	1/n		
Palaviduthi	4.1181	0.924	0.924x+1.4154	0.46
Pelamedu	3.6917	1.0601	1.0601x+1.3061	0.54
Irugur	3.5837	0.7962	0.7962x+ 1.2764	0.46
Vyolagam	3.5169	1.0071	1.0071x+ 1.2576	0.53

Fig 1: Sorption and Desorption behaviour of sulphur in Vylogam series (Typic Rhodustalf)
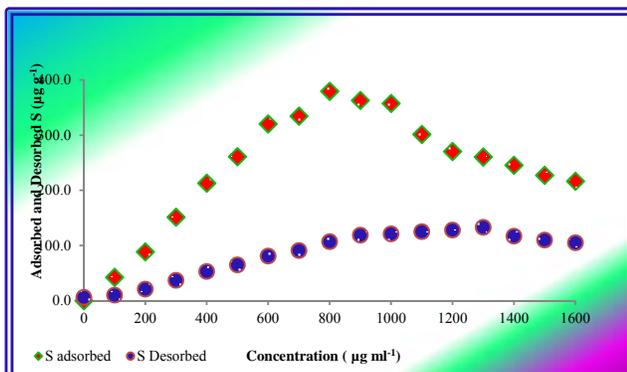

Fig 2: Sorption and Desorption behaviour of sulphur in Palaviduthi series (Typic Haplustalf)
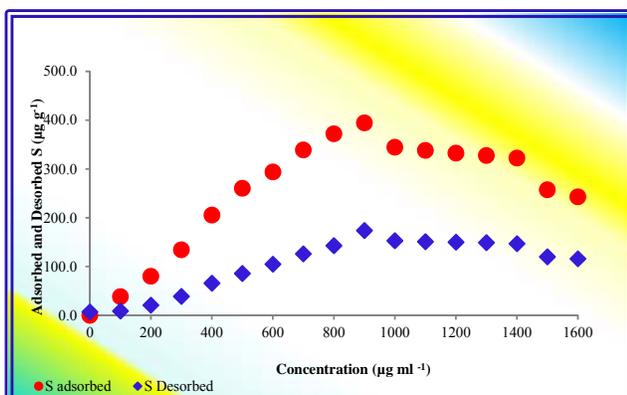

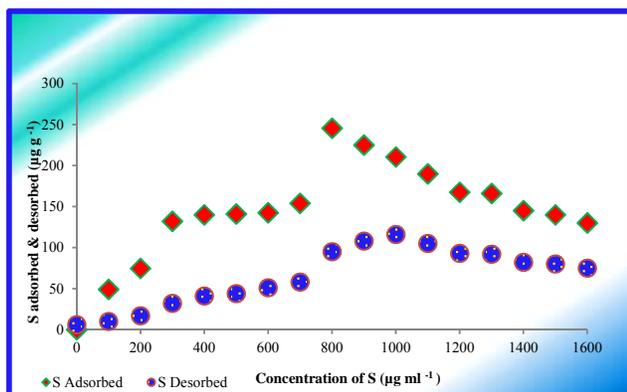**Fig 3:** Sorption and Desorption behaviour of sulphur in Peelamedu series (Typic Haplustert)

Fig 4: Sorption and Desorption behaviour of sulphur in Irugur series (Typic Ustropept)

4. Conclusion

The present results shows that, the S deficiency was main accorded through, the irreversible nature of sulphate sorption capacity largely depends on the pH, amorphous and crystalline Fe and Al content, available phosphorus and organic carbon content, clay, Ca and Mg. The available sulphate content of these soils further moderated the adsorption processes of this anion. However, it can be concluded that the quantitative description of sulphate retention can be used to determine the critical solution concentration of labile sulphate content in these soils for optimum plant growth. So for higher hysteretic and higher sorption capacity soils, more specifically greater sulphur deficient soils, adequate sulphur fertilization (30 kg ha^{-1}) is needed to reduce the sub optimal growth and yield of the pulses in Tamil Nadu.

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