Journal of Applied and Natural Science 8 (4): 1981-1986 (2016)

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Extractability and availability index of sulphur in selected soils of Odisha

Dhaneshwar Padhan[1*], Arup Sen[1] and Pragyan Paramita Rout[2]

[1]Department of Agricultural Chemistry and Soil Science, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur-741235 (West Bengal), INDIA
[2]Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore-641003 (T.N), INDIA
[*]Corresponding author. E-mail: dhaneshwar.padhan@rediffmail.com

Received: January 22, 2016; Revised received: July 20, 2016; Accepted: November 2, 2016

Abstract: We aimed to evaluate the extractability of two reagents viz.0.15% $CaCl_2$ & 0.01M Ca (H_2PO_4) for sulphur along with the sulphur availability index of mixed red and black soils. For this 86 soil samples were collected from mixed red and black soil regions and extracted with these solutions. Results showed that both these solutions extracted nearly similar amount of sulphur in black soils while the Ca phosphate solution extracted higher amount (13.9mgkg^{-1}) compared to Ca chloride solution (11.5mgkg^{-1}) in red soil regions. Considering all the 86 soil samples tested, there was an excellent correlation between the extractable sulphur, the highest correlation being reported from black soils (R^2= 0.97). Sulphur availability index was found to be higher in black soils (mean 6.6) compared to red soils (mean 5.1). Also the content of adsorbed sulphur was found to be high in red soils (2.4mgkg^{-1}) compared to black soils (1.5mgkg^{-1}). Correlation matrix and regression equations (Ca phosphate S= 5.00+ 0.77 Ca chloride S) were worked out between the extractable sulphur and soil properties to justify the results.

Keywords: Adsorbed sulphur, Ca chloride, Ca phosphate, Extractable sulphur, Regression equation, Sulphur Availability index

INTRODUCTION

Sulphur (S) is one of the essential secondary macronutrient elements required for optimum growth, metabolism and development of all plants and is rightly called as the fourth major plant nutrient (Rathore *et al.,* 2015). It plays vital role in different physiological and biochemical functions in plants. However, the non-availability of S in adequate amount in soils leads to reduction in crop yields. Sulphur deficiency in crops has become increasingly widespread all over the world soils (Zhao *et al.,* 2002; Scherer, 2009), the reason being the nutrient management strategies mainly depended on application of NPK fertilizers, ignoring the replenishment of other nutrients through fertilizers or organic sources (Sahrawat *et al.,* 2009) . Apart from that, progressively higher removal of sulphur owing to high production level led to appearance of sulphur deficiency (Tandon, 2011). Such soil S deficiency necessitates the application of S-fertilizer based on soil test values in order to harvest a good crop yields (Pareek, 2007) as supply of S through the application of manure is generally meagre because these resources are sparingly used and in small quantities (Rego *et al.,* 2003). In soils, S mostly remains in organic combination, constituting more than 95% (Wang *et al.,* 2006) of total sulphur. Sulphate-S is the form plants prefer to uptake, which availability depends upon the mineralization of organic S in soils. Although, mineralization of organic S is solely a microbes mediated process, the availability of sulphate is controlled by pH, organic carbon and clay content of soils through some adsorption-desorption mechanism. Soils low in pH and high clay content bind the sulphate-S on the edge of colloidal matrix and plants find it difficult to uptake the same from the exchange sites. The adsorption of sulphate on clay matrix varies with the soil types. Red soils adsorb more sulphate-S than black soils possibly because of lower pH and high content of oxides of iron and aluminium (Ghosh and Dash, 2012). High organic carbon content of black soils supply adequate amount of S in soils though available S content of black soils of Indore were found to be below the critical limit (Padhan, 2014). Sulphur uptake and assimilation varies with crop species, the higher amount required by oilseeds while cereals have least requirement. However, the rate of uptake depends upon the availability of S in the plant root zone during the active growing stage of crops as its availability in soils for plant uptake is mostly influenced by various soil properties (Haneklaus *et al*., 2002; Biswas *et al*., 2003).

Various methods have been proposed to evaluate the amounts of soil S available for plant uptake (Mc Grath & Zhao, 1994). Most of the methods for soil S testing involve extraction of soil with a weak salt solution viz. 0.15% $CaCl_2$ (Williams and Steinbergs, 1959) and 0.01M $Ca(H_2PO_4)$ solution (Fox *et al.,* 1964). Phosphate containing solution performs better in soils where adsorption of sulphate-S is predominant (viz. soils high in oxides of Fe & Al having acidic soil pH) as phosphate displaces the sulphate ion from exchange complex in to solution and calcium ion flocculates the soil colloids (Fox *et al.,* 1964). It is therefore important

ISSN : 0974-9411 (Print), 2231-5209 (Online)

that the factor controlling the extractability of different reagents for S in soils are well understood in order to predict the availability of S. Though, S availability in soil is regulated by various soil properties, several S availability indices have been used as an indicator of plant available S. Sulphur Availability Index (SAI) proposed by Donahue *et al.* (1977) is one such index. According to this concept, if a soil contains sulphate-S just above the critical limit with low organic matter content, then it will not be enough to consider the soil sufficient in available sulphur, since low organic matter content can't support the inorganic S fraction in case of any depletion.

Knowledge regarding the sulphur supplying capacity and the influence of soil properties on its availability in mixed red and black soils of Bargarh district of Odisha is meagre (Padhan, 2014; Padhan *et al.,* 2016). Therefore, the objective of the study were to determine the S supplying capacity of mixed red and black soils and also their Sulphur Availability Index (SAI) in the study areas.

MATERIALS AND METHODS

Eighty six (86) surface soil (0-20cm) samples were collected from mixed red and black soil region of Bargarh district of Odisha where the red soils occupied slightly higher elevation compared to black soils. The study area (fig.1) is having an elevation of 189.3m above the msl. The soils were slightly coarser in texture with moderate clay content. The samples were air dried, ground to pass through 2 mm sieve and stored in polythene bags in a dry place. Soil pH was estimated in soil: 0.01M $CaCl_2$ solution as described by Jackson (1973). Soil organic carbon content was determined by Walkley and Black method (1934). Extractable sulphur was determined by extracting soils with 0.15% $CaCl_2$ solution (Williams and Steinbergs, 1959) and 0.01M Ca (H_2PO_4) solution (Fox *et al.,* 1964). Phosphate containing extractant was used to extract both water soluble and adsorbed sulphate while the Cl^- based extractant was used to extract mainly water soluble sulphate. Sulphur in the extract was determined turbidimetrically (Chesnin and Yien, 1950). Sulphur Availability Index (SAI) was calculated by the formula proposed by Donahue *et al.* (1977) as outlined by Basumatary and Das (2012).

SAI = (0.4 × $CaCl_2$ extractable $SO_4^{=}$ in mg kg^{-1}soil) + % organic matter

Simple correlation was worked out relating extractable S with SAI and soil properties by standard statistical methods.

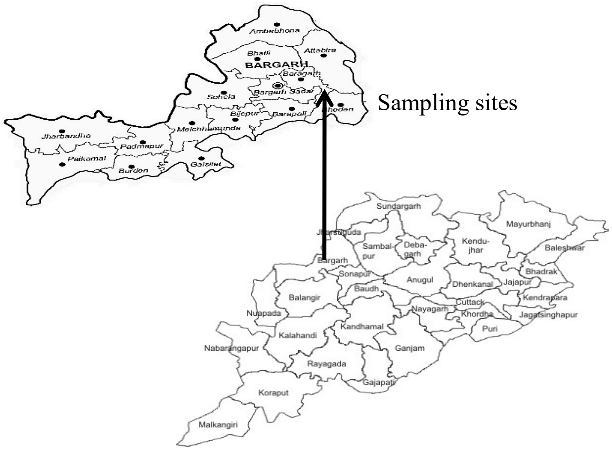

Fig. 1. *Map showing location of sampling sites.*

RESULTS AND DISCUSSION

Soil properties: The prominent soils of the district are mixed red and black type with the former occupying the upper topographic position while the later occurs in lower topographic regions. The results on some of the soil properties revealed that the soil pH ranged from 4.91 to 6.47 with average of 5.86 in red soil regions indicating that the soils were moderately acidic to slightly acidic while the soils of black soil region indicated that the soils were slightly acidic to neutral in reaction which ranged from 6.52 to 7.59 with mean 7.03 (Table 1& 2). Occurrence of such low pH values in the red soil regions could be due to the leaching of bases from the upper topographic position leaving behind the oxides of iron and aluminium which gives lower pH values to the soils (Sehgal, 2012). Black soils have high pH which may be due to presence of high exchangeable cations on the exchange complex and may be due to calcareousness (Kaushal *et al.,* 1986) which in turn is controlled by topography and physiographic position. Accumulations of basic cations especially Ca and Mg in the low lying regions and their subsequent concentration leads to high pH values of the black soils. Similar reports were also observed in the low land soils of some villages of Nimapara block under the coastal plain agro-climatic zone of Odisha (Satpathy *et al.,* 2015). Padhan *et al.* (2015) found that the rice soils of the study regions were acidic to neutral in reaction. The soil organic carbon (SOC) content of red soils were found be low with average of 2.8 g kg^{-1} while that of black soil regions were found to be well above the low status of SOC content (mean 4.6 g kg^{-1}). The magnitude of SOC content in black soils of Bargarh agreed with the findings of Padhan *et al.* (2015). Black soils contain high organic carbon as compared to red soil (Thakrey *et al.,* 2012). High SOC content in black soils compared to red soils could be due to higher rate of oxidation of organic matter in the later owing to its occurrence in the upper topographic position while the high clay content of former possibly make some clay-organic complex and lowering its rapid decomposition.

Extractable sulphur content in soils: Amount of sulphur extracted by several extractants varies with the soil properties. The results of extractable S in soil samples extracted by Ca chloride (0.15% $CaCl_2$ solution) and Ca phosphate (0.01M Ca H_2PO_4) reagents were presented in table 3& 4. Results showed that high amount of Ca phosphate extractable S compared to Ca chloride was found in both the red and black soils. However, the amount of S extracted by both these extractants in black soils was not much differing compared to red soils. It is expected that phosphate containing solution extract S from both the adsorbed and readily available pool while Ca chloride

Table 1. Soil organic carbon (SOC) content and pH of red soils (Mean values of three replications).

Sample No.	pH	Soil organic carbon (gkg^{-1})
1	4.93± 0.05	1.4± 0.21
2	5.10± 0.02	2.1± 0.10
3	5.06± 0.05	2.3± 0.20
4	5.28± 0.06	2.2± 0.10
5	5.61± 0.04	3.1± 0.06
6	5.04± 0.06	2.6± 0.15
7	4.91± 0.03	2.5± 0.12
8	6.27± 0.04	1.4± 0.12
9	6.34± 0.06	1.7± 0.25
10	5.86± 0.07	2.6± 0.15
11	6.31± 0.04	3.3± 0.21
12	5.82± 0.06	3.4± 0.26
13	5.67± 0.05	2.7± 0.26
14	5.82± 0.05	2.5± 0.36
15	5.90± 0.02	2.7± 0.20
16	5.29± 0.05	2.3± 0.10
17	6.27± 0.05	3.1± 0.25
18	6.47± 0.09	3.2± 0.20
19	6.14± 0.03	3.5± 0.30
20	6.19± 0.03	3.6± 0.20
21	6.22± 0.02	3.5± 0.20
22	6.22± 0.03	3.4± 0.31
23	6.03± 0.04	3.1± 0.15
24	6.09± 0.04	3.3± 0.15
25	5.34± 0.10	3.5± 0.30
26	5.46± 0.05	3.2± 0.25
27	5.53± 0.04	3.6± 0.15
28	5.84± 0.06	2.8± 0.25
29	5.86± 0.04	3.1± 0.25
30	6.16± 0.03	2.9± 0.15
31	6.16± 0.06	2.5± 0.35
32	6.27± 0.05	2.5± 0.12
33	6.42± 0.05	2.6± 0.42
34	6.21± 0.05	3.1± 0.26
35	6.05± 0.07	2.8± 0.10
36	6.30± 0.05	2.7± 0.17
37	6.08± 0.04	2.6± 0.20
38	6.25± 0.04	3.0± 0.20
39	6.39± 0.03	2.7± 0.21
40	5.66± 0.04	2.4± 0.26
41	5.91± 0.06	2.7± 0.20
42	5.42± 0.04	2.4± 0.26
Range	4.91- 6.47	1.4- 3.6
Mean	5.86	2.8
SD	0.45	0.53

extract only the readily available pool of S in soils. This happens to be due the greater anion replacing capacity of phosphate ion compared to chloride ion. High content of oxides of Fe and Al in red soils (Ghosh and Dash, 2012) and low pH values exhibit positive charge on edge and surface of soil colloids which suppose to bind the sulphate ion rendering its availability for plant uptake. Soil pH greatly affects the extraction of S from soil by Ca chloride and Ca phosphate. When soil pH is in the neutral and alkaline range, soil particles predominantly carry negative charges and sulphate ions are present in soil solution, leading to extraction of similar amounts of S by both Ca chloride and Ca phosphate (Sahrawat *et al.,* 2009).The high extractable sulphur content in black soils could be due to the high organic carbon content of such soils. Contrary to that despite of having high content of clay in black soils there was no significant adsorption of sulphate compared to red soils because pH of soils played the dominant role in controlling the adsorption-desorption of sulphate ion in soils. Adsorbed S usually found in soils

Table 2. Soil organic carbon (SOC) content and pH of black soils (Mean values of three replications).

Serial No.	pH	Soil organic carbon (gkg^{-1})
1	6.58± 0.09	2.7± 0.21
2	7.10± 0.07	2.3± 0.20
3	7.49± 0.04	2.6± 0.25
4	6.96± 0.06	3.3± 0.20
5	6.58± 0.06	5.2± 0.35
6	6.57± 0.06	5.7± 0.50
7	6.83± 0.05	2.5± 0.31
8	6.61± 0.04	5.5± 0.23
9	6.79± 0.04	3.5± 0.36
10	6.62± 0.06	3.5± 0.35
11	7.16± 0.05	4.2± 0.30
12	7.16± 0.06	4.5± 0.35
13	7.31± 0.05	4.9± 0.20
14	7.40± 0.05	4.7± 0.45
15	7.54± 0.09	5.5± 0.40
16	7.24± 0.05	4.4± 0.20
17	7.29± 0.06	3.5± 0.30
18	7.17± 0.05	3.5± 0.21
19	7.39± 0.04	3.6± 0.30
20	6.88± 0.04	5.4± 0.20
21	6.86± 0.08	6.2± 0.30
22	6.85± 0.07	3.5± 0.20
23	6.52± 0.07	5.3± 0.17
24	6.60± 0.04	5.2± 0.17
25	6.67± 0.07	6.4± 0.15
26	6.84± 0.06	5.5± 0.40
27	6.89± 0.03	5.5± 0.31
28	6.96± 0.07	4.3± 0.26
29	6.84± 0.07	4.6± 0.26
30	7.37± 0.05	4.6± 0.35
31	7.59± 0.05	5.5± 0.35
32	7.34± 0.12	4.9± 0.35
33	7.19± 0.06	5.1± 0.25
34	7.38± 0.04	6.1± 0.25
35	7.59± 0.03	5.4± 0.25
36	7.21± 0.03	4.8± 0.31
37	7.52± 0.04	4.5± 0.40
38	6.90± 0.02	4.4± 0.15
39	6.79± 0.07	4.2± 0.23
40	6.64± 0.06	4.1± 0.25
41	6.65± 0.10	3.6± 0.35
42	6.82± 0.06	5.5± 0.31
43	7.19± 0.07	5.2± 0.25
44	7.20± 0.06	4.8± 0.30
Range	6.52- 7.59	2.3- 6.4
Mean	7.03	4.60
SD	0.32	1.01

Table 3. Extractable S (mg kg^{-1}) extracted with 0.15% $CaCl_2$ (Ca chloride S) and 0.01M Ca (H_2PO_4) (Ca phosphate S) solutions in 42 soil samples of red soil regions.

Sample No.	0.15% $CaCl_2$ extractable S	0.01M Ca (H_2PO_4) extractable S
1	9.3± 0.15	14.9± 0.31
2	10.3± 0.17	14.6± 0.25
3	10.6± 0.15	14.4± 0.30
4	11.4± 0.15	13.6± 0.26
5	11.6± 0.36	13.5± 0.30
6	10.4± 0.25	14.5± 0.26
7	9.6± 0.47	15.5± 0.36
8	11.5± 0.30	13.1± 0.15
9	11.5± 0.30	12.4± 0.25
10	12.5± 0.40	13.3± 0.21
11	12.5± 0.26	13.5± 0.31
12	11.9± 0.35	13.3± 0.26
13	12.4± 0.45	14.4± 0.25
14	14.0± 0.21	15.6± 0.21
15	11.7± 0.40	14.6± 0.35
16	11.2± 0.25	14.5± 0.31
17	11.0± 0.31	12.5± 0.36
18	11.5± 0.32	13.2± 0.30
19	12.5± 0.25	13.5± 0.31
20	12.4± 0.31	13.7± 0.26
21	12.2± 0.36	14.5± 0.31
22	11.6± 0.20	14.2± 0.36
23	11.6± 0.32	13.5± 0.30
24	12.9± 0.35	15.5± 0.23
25	12.5± 0.40	13.5± 0.31
26	11.6± 0.31	12.4± 0.30
27	11.2± 0.35	12.8± 0.31
28	10.6± 0.23	12.6± 0.35
29	11.5± 0.31	13.5± 0.31
30	10.6± 0.06	14.6± 0.30
31	10.5± 0.20	12.5± 0.35
32	11.5± 0.31	16.4± 0.45
33	12.8± 0.36	15.3± 0.50
34	11.8± 0.25	13.6± 0.36
35	12.8± 0.35	14.5± 0.31
36	10.9± 0.31	12.5± 0.36
37	10.5± 0.25	13.5± 0.31
38	10.5± 0.31	12.1± 0.25
39	12.2± 0.31	14.6± 0.25
40	11.5± 0.26	14.6± 0.25
41	11.7± 0.31	14.5± 0.30
42	11.8± 0.25	15.5± 0.31
Range	9.3- 14.0	12.1- 16.4
Mean	11.5	13.9
SD	0.92	1.02

with low pH values and high content of oxides of Fe and Al. Results (table. 6) showed that the red soils were found to be high in adsorbed S (average 2.4 mg kg^{-1}) compared to black soils (average 1.5 mg kg^{-1}). The possible reason for such occurrence could be due to the low pH values of red soils. Sulphate adsorption has been attributed to a pH dependent positive charge typical of acidic soils (Scherer, 2009). In neutral to alkaline soils, there was no appreciable adsorption of sulphur noticed (Shahsavani *et al.,* 2006; Cui *et al.,* 2006).

The regression analysis between the values of S extracted by Ca chloride ($CaCl_2$–S) and Ca phosphate (Ca phosphate–S) reagents for all the 86 soil samples studied showed that they were highly significantly correlated (R^2= 0.82, n = 86) (table. 7). Although highly significant correlation coefficient between Ca chloride and Ca phosphate extractable S was found in black soils (R^2= 0.97), Ca phosphate extracted higher amount of S than Ca chloride solution. Sahrawat *et al.* (2009) also reported that Ca phosphate could extract higher amount of S compared to Ca chloride in low pH soils.

Table 4. Extractable S (mg kg^{-1}) extracted with 0.15% $CaCl_2$ (Ca chloride S) and 0.01M Ca (H_2PO_4) (Ca phosphate S) solutions in 44 soil samples of black soil regions.

Serial No.	0.15% $CaCl_2$ extractable S	0.01M Ca (H_2PO_4) extractable S
1	15.2± 0.40	16.4± 0.95
2	11.9± 0.40	13.8± 0.46
3	15.2± 0.35	16.5± 0.40
4	16.2± 0.35	17.6± 0.86
5	11.9± 0.50	13.6± 1.12
6	14.2± 0.30	16.2± 0.70
7	13.8± 0.35	15.6± 1.15
8	13.6± 0.36	14.5± 0.70
9	14.5± 0.31	15.8± 0.75
10	16.4± 0.23	17.8± 0.96
11	14.5± 0.26	15.7± 0.64
12	15.6± 0.32	16.6± 0.76
13	16.5± 0.25	17.7± 0.58
14	16.3± 0.40	17.5± 1.05
15	15.9± 0.35	16.9± 0.38
16	14.5± 0.25	15.9± 0.81
17	13.6± 0.35	14.9± 0.46
18	12.7± 0.26	14.1± 0.66
19	12.8± 0.46	14.5± 0.85
20	16.4± 0.26	17.9± 0.46
21	13.9± 0.57	15.9± 0.74
22	16.4± 0.56	17.6± 1.01
23	15.5± 0.32	16.9± 0.82
24	14.6± 0.32	15.9± 0.50
25	16.5± 0.31	17.5± 0.31
26	18.3± 0.21	19.7± 0.58
27	19.2± 0.40	20.4± 0.61
28	15.5± 0.31	17.1± 0.31
29	16.7± 0.46	17.9± 0.55
30	14.6± 0.17	16.2± 0.45
31	17.3± 0.46	19.0± 1.10
32	18.4± 0.46	19.8± 0.85
33	16.8± 0.66	18.4± 0.49
34	18.7± 0.44	20.0± 0.53
35	14.2± 1.19	16.2± 0.60
36	14.3± 0.51	15.2± 0.65
37	13.4± 0.74	14.9± 0.81
38	17.6± 0.51	19.4± 0.53
39	17.6± 0.25	19.4± 0.87
40	15.2± 0.89	17.0± 0.50
41	16.6± 0.35	17.9± 0.56
42	13.9± 0.53	15.8± 0.60
43	14.4± 0.46	15.7± 0.58
44	15.8± 0.60	17.3± 0.46
Range	11.9- 19.2	13.6- 20.4
Mean	15.4	16.8
SD	1.74	1.69

Sulphur availability index: The available sulphur

Table 5. Correlation coefficients of extractable S (Ca chloride & Ca phosphate S) and SAI with soil properties and Adsorbed S in soils of both red and black soil regions.

Soil type	Correlation coefficient of 15% $CaCl_2$ extractable S with			Correlation coefficient of 0.01M $Ca(H_2PO_4)$ extractable S with			Correlation coefficient of SAI with			
	pH	Organic C	Adsorbed S	pH	Organic C	Adsorbed S	pH	Organic C	Adsorbed S	0.01M Ca (H_2PO_4) extractable S
Red soils	0.415**	0.386**	-0.589**	-0.254	-0.207	-0.453**	0.433**	0.581**	-0.623**	0.117
Black soils	0.012	0.293	-0.254	0.05	0.296	-0.087	0.011	0.409**	-0.234	0.980**

** Correlation is significant at the 0.01 level (2-tailed); * Correlation is significant at the 0.05 level (2-tailed)

Table 6. Range and mean values of SAI and Adsorbed S in soils of red and black soil regions.

Soil type	SAI		Adsorbed S (mg kg^{-1})	
	Range	Mean	Range	Mean
Red soils	4.0-6.0	5.1	0.8-5.9	2.4
Black soils	5.0-8.2	6.6	0.9-2.1	1.5

fraction, which consists largely of easily extractable sulphate in soils, is the immediate supplier of S to plant roots. Calcium chloride and mono calcium phosphate solutions are commonly used extractants for predicting the available S content in soils. But in the present study we considered the Ca chloride extractable S as available S. However, only the Ca chloride extractable S may not be adequate to assess the sulphur availability, as the availability is governed by a number of soil properties (Patra *et al*., 2012). The shortcoming can be overcome to certain extent by using the Sulphur Availability Index as a key to assess the available S status in soils (Basumatary and Das, 2012). Based on the values of SAI, the red soils were found to be deficient while the black soils were of deficient to medium status. The SAI values of red soils ranged from 4.0-6.0 with an average of 5.1 and in black soil it varied from 5.0-8.2 with an average of 6.6 (table. 6). As the SAI index is a function of Ca chloride extractable S and organic matter content of soils, black soils being high in both Ca chloride extractable S and organic matter content showed higher SAI values compared to red soils. Patra *et al.* (2012) found that majority of the soil samples tested of red and lateritic zone of West Bengal were having SAI values below 6.0 and categorized as low status.

Relationship of extractable S status with soil properties: Correlation coefficients of some important soil characteristics with Ca chloride and Ca phosphate extractable S and SAI were presented in table.5. Calcium chloride extractable S maintained significant positive correlation with SOC (0.386**), pH (0.415**) and significant but negative correlation with adsorbed S (-0.589**) in red soil regions while no such relationship were observed in black soils regions. Soil organic carbon content of both red and black soils showed significant and positive agreement with SAI while the adsorbed S maintained significant negative correlation with SAI of red soil regions. Soil pH maintained significant positive correlation with SAI in red soils.

Table 7. Relationships between S extracted by 0.15% $CaCl_2$ (Ca chloride S) and 0.01M Ca (H_2PO_4) (Ca phosphate S) solutions in red and black soils.

Soil type	Number of samples	Regression equation	Correlation coefficient (R^2)
Red	42	Ca phosphate S= 11.45+ 0.21 Ca chloride S	0.037
Black	44	Ca phosphate S= 2.09+ 0.95 Ca chloride S	0.97
Red-Black	86	Ca phosphate S= 5.00+ 0.77 Ca chloride S	0.82

Conclusion

The results of the study emphasized the importance of soil properties on the extractability different reagents for S in soils. Both the reagents extracted more or less similar amounts of S in black soils where as in case of red soils Ca phosphate extracted somewhat high amount of S compared to Ca chloride solution. However, the amount of available S was found in the critical region suggesting the need of external supplementation through fertilizers or manures. Application of organics along with chemical fertilizers will be beneficial since it will support the inorganic fraction of S in case of severe depletion during crop growth and also improve the sulphur availability index (SAI) in soils.

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