Extractable Fractions of Sulphur in Major Soils of India

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Abstract
We evaluated four different methods viz. 0.15% $CaCl_2$, 0.01M $Ca(H_2PO_4)_2$, water soluble and heat soluble for their extractability of different fractions of sulphur (S) in red, alluvial and black soils of India. Twenty composite soil samples (0-0.2m depth) from each soil types were collected and analyzed for their chemical properties and extractable S fractions. Result showed that the extractability of the different methods followed the order of $Ca(H_2PO_4)_2$-S > Heat-S > $CaCl_2$-S > Water-S irrespective of soil types. Black soils witnessed with high amount of extractable fractions of sulphur compared to red and alluvial soils. The correlation and regression analysis between soil properties and extractable fractions as well as among the extractable fractions of sulphur was worked out to evaluate the methods. A strong relationship between organic carbon content and heat soluble S was observed irrespective of soil types.

Keywords: Sulphur fraction, Heat soluble sulphur, Water soluble sulphur, $CaCl_2$ extractable sulphur and $Ca(H_2PO_4)_2$ extractable sulphur

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Introduction

Sulphur (S) is a secondary nutrient element essential for the growth and development of plant and animal species. Although, the requirement of sulphur is less compared to other primary nutrients, the uptake in some plants is equivalent with that of phosphorus. It plays a key role in synthesis of S-containing amino acids (cysteine, cystine and methionine), that are essential components of plant protein and comprises a major part of sulphur in plant. The major source of sulphur for plant nutrition is soil besides chemical fertilizers and organic manures application. However, in recent years S deficiency has become increasingly widespread in soils of India, especially coarse textured alluvial soils, red and lateritic soils, leached acidic soils as well as soils with low organic matter content. The reasons for such occurrence are mainly due to decreased S inputs from the atmosphere and fertilizers (replacement of SSP by DAP), low organic matter content in soils and inadequate addition of organic manures followed by crop removal with high yielding varieties and intensive perturbation, and adsorption of S in acid soils. Increased S deficiency has led to a greater need for soil testing and plant analysis to diagnose whether application of fertilizer S is necessary. Soil and plant analysis is usually a reliable tool in the diagnosis of S deficiency [1].

Sulphur in soils exists from -2 to +6 oxidation states forming various stable and unstable compounds with other elements and thus, regulating its availability for crop nutrition. Considering the relative extractability and plant availability, the major forms of S in soils are – sulphate (SO_4-S) ions in the soil solution, sulphate ions adsorbed by inorganic colloids, inorganic compounds in insoluble forms (pyrite and marcasite (FeS_2), pyrrhotite ($Fe_{1-x}S$) and chalcopyrite ($CuFeS_2$), and organic S compounds (Thiols, disulfides, polysulfides and ester sulphates). The sulphate ion is primarily adsorbed by clay minerals and Fe/Al oxides and adsorption increases with decrease in pH below 5.5 [2]. Several extractants have been tested to evaluate the S availability for plant uptake. Suitability of extractant for estimation of different forms of S in soil is regulated by type of soil and the most important is the kind of sulfur fractions and their proportionate contribution [3]. For soils of diversified physical and chemical characteristics, a common extractant cannot be referred for predicting the S availing ability of all the soils and there is great need to find out the promising extractants which may give dependable results for a definite group of soils [4]. In view of this, a study on the amount of S extracted by different extractants and their relation with soil properties in Alfisols, Inceptisol and Vertisol were undertaken. The present study aimed for selection of a promising extractant for estimating available S in major soil groups of India.

Materials and Methods

Four soil series from each three major soil types in India viz. red, alluvial and black correspond to Alfisol, Inceptisol and Vertisol respectively were selected for the present study. Surface soils (0-0.20m) were collected from five

different sites from each four soil series in three replicates. Collected soil samples were air-dried, grinded and mixed thoroughly and then passed through 2mm sieve for analysis of physic-chemical and chemical properties.

Collected soils were analyzed for pH_{water}, pH_{CaCl_2}, electrical conductivity as described by [5]. Oxidizable organic carbon and available nitrogen were determined by acid digestion method [6] and alkaline permanganate oxidation method [7] respectively. Extractable sulphur was determined by extracting soils with 0.15% $CaCl_2$ solution [8] and 0.01M $Ca(H_2PO_4)_2$ solution [9]. Water soluble sulphur and heat soluble sulphur were (with 1% NaCl) determined following the method outlined by Williams and Steinbergs in 1959 [8]. Sulphur in the extract was determined turbidimetrically [10]. Details of the methods used to measure sulphur were presented on **Table 1**.

Simple correlation matrix was worked out between soil properties and different forms of sulphur. Also linear regression equations have been drawn between different methods of sulphur estimation.

Table 1 Details of the methods used for estimation of extractable S in the selected soils

Extractant	Soil: extractant ratio	Shaking time (min)	References
0.01M $Ca(H_2PO_4)_2$	1:5	30	[9]
0.15% $CaCl_2$	1:5	30	[8]
Water soluble S	1:5	30	[8]
Heat soluble S	5:33	30	[8]

Results
Soil properties

Results depicted in **Table 2-4** showed a wide variation in soil properties. The pH ranged from 5.1 to 7.1 in red soils having acidic in nature, 7.4 to 8.6 in black soils showing various degrees of alkalinity. However, the alluvial soils showed slightly acidic to neutral pH ranging from 5.5 to 8.4.

Table 2 Chemical properties of the selected soil series of red soil regions (Mean value of three replications)

Series	Sites	pHw	pHCa	ΔpH	EC (dS m^{-1})	OC (%)	N (g kg^{-1})
Kusmi	1	5.3	5.1	0.14	0.21	0.71	0.116
	2	5.6	5.5	0.11	0.23	0.77	0.161
	3	5.9	5.8	0.11	0.20	0.70	0.145
	4	5.8	5.6	0.16	0.23	0.77	0.151
	5	5.1	5.0	0.12	0.21	0.77	0.130
Gangalghati	1	6.3	6.1	0.13	0.14	0.47	0.085
	2	5.7	5.4	0.23	0.21	0.75	0.088
	3	6.3	6.2	0.13	0.14	0.50	0.099
	4	5.3	5.2	0.11	0.12	0.41	0.067
	5	5.8	5.7	0.10	0.25	0.79	0.137
Khejuria	1	6.2	6.1	0.15	0.17	0.61	0.114
	2	6.6	6.5	0.13	0.23	0.83	0.124
	3	5.6	5.5	0.12	0.13	0.46	0.057
	4	5.4	5.3	0.11	0.21	0.75	0.139
	5	5.6	5.5	0.07	0.17	0.62	0.127
Rangamati	1	5.9	5.8	0.13	0.14	0.46	0.070
	2	6.7	6.6	0.14	0.17	0.62	0.098
	3	7.1	6.8	0.31	0.14	0.48	0.084
	4	5.9	5.7	0.13	0.11	0.37	0.069
	5	6.5	6.4	0.13	0.19	0.66	0.106
	Range	5.1-7.1	5.0-6.8	0.07-0.23	0.11-0.25	0.37-0.83	0.057-0.161
	Mean	5.92	5.78	0.14	0.18	0.63	0.11
	SD	0.53	0.51	0.05	0.04	0.15	0.03
	CV	9.01	8.81	37.14	23.16	23.43	28.35

The delta change in pH (ΔpH) was more or less equal in red (0.14) and black (0.14) soil but comparatively lesser in alluvial (0.12) soil. There was not much difference (0.18, 0.19 and 0.20 dSm^{-1} in red, alluvial and black soil respectively) in the value of electrical conductivity among the tested soils. Black soils were found to be high in terms of oxidizable organic carbon content followed by alluvial and red soils. It ranged from 1.18 to 2.32, 0.72 to 2.69 and

0.37 to 0.83 % in black, alluvial and red soils with a mean value of 1.67, 1.55 and 0.63 % respectively. Similar trend was observed for available nitrogen content in soils being high in black soils followed by alluvial and red soils.

Table 3 Chemical properties of the selected soil series of alluvial soil regions (Mean value of three replications)

Series	Sites	pHw	pHCa	ΔpH	EC (dS m^{-1})	OC (%)	N (g kg^{-1})
Kusmi	1	7.5	7.4	0.15	0.15	2.69	0.32
	2	7.3	7.2	0.11	0.17	2.29	0.29
	3	7.6	7.4	0.21	0.15	2.55	0.28
	4	6.4	6.2	0.20	0.18	1.80	0.20
	5	6.8	6.6	0.19	0.17	2.26	0.23
Gangalghati	1	6.2	6.2	0.09	0.26	1.04	0.10
	2	5.8	5.7	0.09	0.25	0.72	0.09
	3	5.6	5.6	0.07	0.17	1.10	0.11
	4	5.5	5.3	0.17	0.13	0.82	0.08
	5	6.2	6.1	0.13	0.27	0.85	0.09
Khejuria	1	8.4	8.3	0.09	0.17	2.18	0.29
	2	7.9	7.8	0.10	0.19	1.76	0.29
	3	7.8	7.6	0.12	0.15	1.91	0.35
	4	7.5	7.4	0.11	0.19	1.57	0.20
	5	7.7	7.7	0.08	0.25	1.51	0.19
Rangamati	1	6.4	6.3	0.02	0.17	1.73	0.16
	2	6.7	6.6	0.09	0.20	0.97	0.12
	3	7.2	7.1	0.11	0.24	0.86	0.10
	4	6.3	6.2	0.08	0.14	1.17	0.14
	5	6.6	6.5	0.14	0.22	1.15	0.14
	Range	5.5-8.4	5.3-8.3	0.02-0.21	0.13-0.27	0.72-2.69	0.09-0.35
	Mean	6.87	6.76	0.12	0.19	1.55	0.19
	SD	0.82	0.82	0.05	0.04	0.62	0.09
	CV	11.97	12.15	40.89	22.30	40.16	46.82

Table 4 Chemical properties of the selected soil series of black soil regions (Mean value of three replications)

Series	Sites	pHw	pHCa	ΔpH	EC (dS m^{-1})	SOC (%)	Avl. N (g kg^{-1})
Kusmi	1	8.5	8.4	0.06	0.18	1.48	0.23
	2	8.2	8.1	0.12	0.13	1.59	0.35
	3	8.6	8.4	0.14	0.22	1.25	0.19
	4	8.4	8.3	0.13	0.14	1.18	0.15
	5	8.2	8.1	0.17	0.17	1.73	0.29
Gangalghati	1	7.9	7.7	0.22	0.17	1.70	0.23
	2	7.5	7.4	0.12	0.16	1.75	0.29
	3	8.3	8.2	0.10	0.14	1.92	0.22
	4	8.4	8.3	0.10	0.23	1.52	0.18
	5	8.2	8.1	0.13	0.12	1.45	0.21
Khejuria	1	8.6	8.4	0.14	0.29	2.08	0.38
	2	8.1	8.0	0.11	0.22	1.47	0.41
	3	7.5	7.3	0.12	0.25	1.84	0.45
	4	8.3	8.1	0.15	0.21	1.63	0.24
	5	8.2	8.0	0.18	0.30	1.81	0.34
Rangamati	1	8.2	8.0	0.19	0.18	2.32	0.27
	2	7.8	7.7	0.14	0.23	1.48	0.21
	3	7.4	7.3	0.12	0.20	1.75	0.24
	4	7.4	7.3	0.14	0.15	1.61	0.23
	5	8.1	7.9	0.19	0.26	1.87	0.29
	Range	7.4-8.6	7.3-8.4	0.06-0.22	0.13-0.30	1.18-2.32	0.15-0.45
	Mean	8.08	7.95	0.14	0.20	1.67	0.27
	SD	0.37	0.38	0.04	0.05	0.27	0.08
	CV	4.63	4.77	27.38	26.43	16.10	29.73

Table 5 Extractable S (mg kg^{-1}) content in red, alluvial and black soils

Red soil						Alluvial soil					
Series	Sites	CaCl$_2$S	Ca(H$_2$PO$_4$)$_2$ S	Water S	Heat S	Series	Sites	CaCl$_2$S	Ca(H$_2$PO$_4$)$_2$ S	Water S	Heat S
		(mg kg $^{-1}$)						(mg kg $^{-1}$)			
Kusmi	1	4.76	17.57	2.93	5.39	Balarampur	1	21.09	19.70	15.43	28.42
	2	6.32	13.54	4.56	6.21		2	18.74	22.85	15.58	19.01
	3	7.68	14.64	5.56	5.10		3	22.96	20.95	25.97	25.10
	4	6.92	13.93	5.15	6.52		4	14.14	31.36	10.66	19.00
	5	4.35	17.80	3.20	6.43		5	16.41	30.10	14.50	17.21
Gangalghati	1	9.86	11.33	7.16	5.03	Panchopta	1	10.64	31.43	8.84	15.68
	2	6.74	13.90	4.56	6.70		2	6.61	24.89	4.59	9.90
	3	10.08	10.56	8.15	5.57		3	8.38	27.28	7.12	13.65
	4	4.94	16.35	3.11	4.37		4	6.65	29.99	6.17	10.41
	5	7.16	14.15	6.18	7.48		5	10.36	30.54	8.29	12.52
Khejuria	1	9.64	15.63	7.15	5.94	Tehatta	1	21.00	15.87	17.55	21.29
	2	11.33	9.47	8.14	12.18		2	20.22	17.19	18.84	21.33
	3	6.45	14.33	5.65	5.05		3	20.69	14.73	14.92	20.96
	4	5.23	19.68	5.08	6.49		4	19.32	25.27	18.36	18.85
	5	6.41	16.22	5.65	6.17		5	20.56	20.05	16.59	16.29
Rangamati	1	7.19	14.17	5.80	4.66	Narayanapara	1	12.96	28.70	11.19	23.45
	2	11.31	15.86	10.15	6.27		2	15.89	24.48	12.04	13.80
	3	13.20	11.63	12.33	4.54		3	17.18	20.62	16.11	11.10
	4	7.24	14.53	5.88	4.07		4	13.27	29.25	11.90	17.16
	5	11.19	13.65	9.71	6.53		5	13.26	29.50	12.05	17.05
Mean		7.90	14.45	6.31	6.04	Mean		15.52	24.74	13.33	17.61
SD		2.54	2.50	2.44	1.71	SD		5.11	5.44	5.10	4.96
CV		32.19	17.27	38.67	28.32	CV		32.96	21.99	38.24	28.15

Table 5 Continued

Black soil					
Series	Sites	CaCl$_2$S	Ca(H$_2$PO$_4$)$_2$ S	Water S	Heat S
		(mg kg $^{-1}$)			
Jambha	1	15.37	20.98	13.16	15.85
	2	15.64	21.41	13.55	17.34
	3	18.60	22.64	16.97	12.96
	4	17.57	21.71	16.07	13.67
	5	17.26	21.64	14.97	18.86
Linga	1	15.76	21.45	8.15	19.02
	2	15.05	20.93	13.46	19.55
	3	17.99	18.65	15.02	21.26
	4	19.25	22.48	16.33	16.89
	5	14.89	18.87	11.37	16.46
Nimone	1	20.70	23.22	19.16	23.36
	2	16.88	20.47	14.04	16.57
	3	13.64	24.73	11.23	20.34
	4	17.66	22.22	15.55	18.06
	5	16.45	21.40	14.98	21.07
Sawargaon	1	21.36	17.33	19.66	24.15
	2	15.56	19.72	13.64	17.56
	3	17.87	20.57	14.57	18.36
	4	12.62	24.57	11.16	17.98
	5	16.26	21.13	14.65	21.10
Mean		16.82	21.31	14.39	18.52
SD		2.18	1.82	2.70	2.86
CV		12.94	8.56	18.77	15.45

Extractable sulphur

The amount of extractable sulphur varied with soil types and extraction methods used (**Table 5**). The 0.01M $Ca(H_2PO_4)_2$ solution extracted the maximum amount of S and water did the minimum, the mean extractability value of different extractant ranked in the order $Ca(H_2PO_4)_2$.

S > Heat-S > $CaCl_2$-S > Water-S irrespective of soil types. Similarly, irrespective of extraction methods tested, the extractability followed the order of black> alluvial> ordered soils.

The amount of sulphur extracted by 0.15% $CaCl_2$ ranged from 4.35 to 13.20, 6.61 to 22.96, and 12.62 to 21.36 mgkg^{-1} in red, alluvial and black soils with mean value of 7.90, 15.52 and 16.82 respectively. However, the 0.01M $Ca(H_2PO_4)_2$ extractable S was found much higher in all soil compared to other methods, ranging from 9.47 to 19.68, 14.73 to 31.43 and 17.33 to 24.73 mgkg^{-1} in red, alluvial and black soils with mean value of 14.45, 24.74 and 21.31 mgkg^{-1} respectively. Black soils witnessed with high amount of heat soluble sulphur followed by alluvial and red soils with mean value of 18.52, 17.61 and 6.04 mgkg^{-1} respectively. Water soluble sulphur followed similar trend with mean sulphur content of 14.39, 13.33 and 6.31 mgkg^{-1} in black, alluvial and red soils respectively.

The $CaCl_2$-S and Water-S was significantly and positively correlated with pHw and pH$_{CaCl2}$ in all three soil types (**Table 6**). Significant positive correlations between heat soluble sulphur and organic carbon (SOC) and available N in were found all soils. Extractable sulphur fractions showed significant correlation with SOC in inceptisols.

Table 6 Relationship of different fractions of sulphur with soil properties

	CaCl₂-S			Ca(H₂PO₄)₂-S			Water-S			Heat-S		
	Red	Alluvial	Black	Red	Alluvial	Black	Red	Alluvial	Black	Red	Alluvial	Black
pH$_w$	0.98**	0.94**	0.55**	-0.66**	-0.80**	-0.17	0.95**	0.85**	0.51**	0.19	0.50**	-0.18
pH$_{CaCl2}$	0.97**	0.93**	0.53**	-0.65**	-0.80**	-0.16	0.93**	0.83**	0.49**	0.20	.50**	-0.21
EC	-0.16	-0.20	0.20	0.12	0.11	0.34**	-0.19	-0.17	0.29*	0.61**	-0.25	0.32*
SOC	-0.16	0.75**	0.32*	0.15	-0.43**	-0.22	-0.20	0.69**	0.26*	0.69**	0.74**	0.96**
Avl. N	-0.13	0.80**	-0.11	0.16	-0.64**	0.21	-0.17	0.67**	0.004	0.34**	0.66**	0.42**

** Correlation is significant at the 0.01 level; * Correlation is significant at the 0.05 level

Discussion

Because of high rainfall leaching of bases from the upper topographic position occurs leaving behind the concentrate of oxides of iron and aluminium that could lower the pH values in the red soil regions [11]. However, the presence of base enriched zeolite and other minerals in black soils could be the reason for its high pH values. High exchangeable cations on the exchange surface as well as the presence of carbonates of Ca and Mg further increase the pH values in black soil. Higher OC content in black soils followed by alluvial and red soils could be due to higher rate of oxidation of organic matter in red soil owing to its occurrence in the upper topographic position while the higher clay content of black soil possibly make some clay-organic complex and lowering its rapid decomposition. As the carbon content of soils have a positive effect on the magnitude of nitrogen content in soil, black soils witnessed high available nitrogen content compared to alluvial and red soils.

Higher extractable S with 0.01M $Ca(H_2PO_4)_2$ than 0.15% $CaCl_2$ may be attributed to greater anion exhange capacity of phosphate than chloride ion. Due to greater anion exchange capacity, phosphate containing solution can extract S from both the adsorbed and readily available pool [12].

Ease of release of 0.15% $CaCl_2$ and 0.01M $Ca(H_2PO_4)_2$ extractable SO_4^{2-} ion from the exchange surface is mostly dependent on pH dependent positive charge of clay. Presence of higher amount of oxides of Fe and Al in red soil [13, 14] as well as low soil pH exhibits pH-dependent positive charge on edge and surface of soil colloids which ultimately hold higher sulphate ions on exchange surfaces. Thus, red soil exhibited higher $Ca(H_2PO_4)_2$-S compared to others. Similar result reported by Padhan et al. (2016) [15] in red soils of Odisha. At neutral and alkaline pH, soil particles predominantly carry negative charges, thus lowering the amount of clay adsorbed S in alluvial and black soils. Our results were in line with Sahrawat et al., (2009) [16]. The mean $Ca(H_2PO_4)_2$-S was higher in black and alluvial soil compared to red soil and this might be due to higher buffering capacity of alluvial and black soil. Higher heat soluble sulphur content in alluvial and black soils could be due to the high organic carbon content of such soils [17]. Heat soluble S is the labile fraction of organic S that could be expected to be mineralized during subsequent cropping season.

Table 7 Relationships among the extractable fractions of sulphur in soils

Extractants/Methods	Red	Alluvial	Black
$CaCl_2$-S vs $Ca(H_2PO_4)_2$-S	-0.68[**]	-0.71[**]	-0.32[*]
$CaCl_2$-S vs Water-S	0.94[**]	0.91[**]	0.74[**]
$CaCl_2$-S vs Heat-S	0.202	0.56[**]	0.23
$Ca(H_2PO_4)_2$-S vs Water-S	-0.54[**]	-0.61[**]	-0.19
$Ca(H_2PO_4)_2$-S vs Heat-S	-0.30[*]	-0.33[*]	-0.19
Water-S vs Heat-S	0.12	0.52[**]	0.23

[**] Correlation is significant at the 0.01 level
[*] Correlation is significant at the 0.05 level
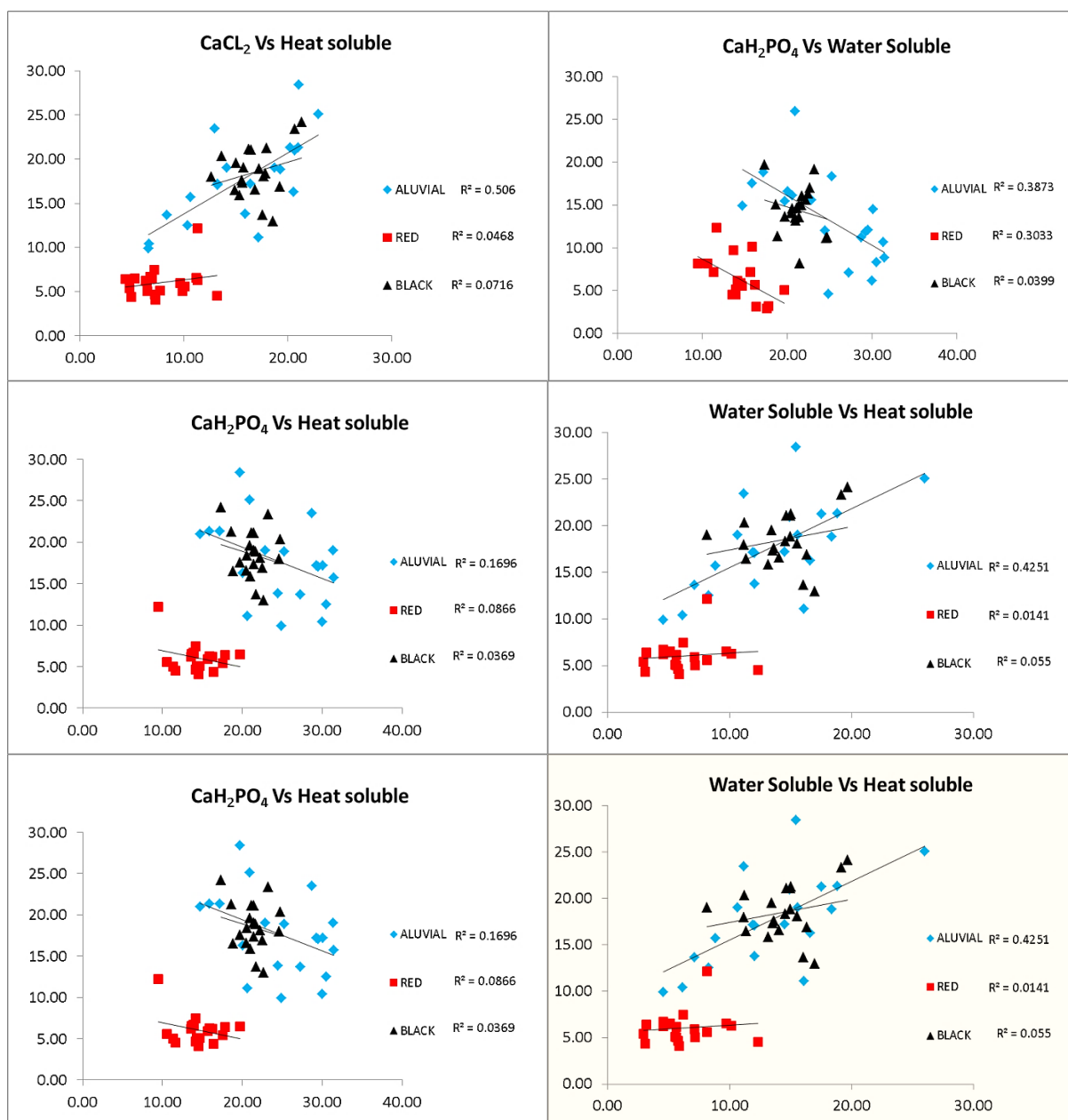
Figure 1 Linear regression analysis between fractions of sulphur in different soils

The correlation and regression analyses between the values of S extracted by all four methods were studied and presented in **Table 7** and **Figure 1**. Result showed that the amount of sulphur extracted by the extractants viz. $CaCl_2$-S vs. Water-S, (for all soil types) and Water-S vs. Heat-S, $CaCl_2$-S vs. Heat-S (only for alluvial soil) were significantly and positively correlated. Whereas, $CaCl_2$-S vs CaH_2PO_4-S and CaH_2PO_4-S vs Water-S extractants were significantly but negatively correlated (Table 7). This negative relation might be due to release of adsorbed sulphur to

the soil solution with the increase in pH of soil [18], as we have got significant positive correlation between pH and $CaCl_2$-S and Water-S, but a significant negative correlation between pH and CaH_2PO_4-S. The strong relationship between OC and heat soluble S irrespective of soil types indicated that the S fraction stemmed from the soil organic carbon content. We found a significant negative correlation in all soil types between $Ca(H_2PO_4)_2$-S vs Heat-S as well as between $Ca(H_2PO_4)_2$-S and oxidizable organic carbon. This observation might be due to inhibition of anion adsorption capacity by soil organic matter, especially organic anions, which tend to bind to the reactive surfaces of Fe and Al minerals [19, 20].

Conclusion

Extractable sulphur content in all the soil types showed wide variations. And as such the extractability was governed by these diverse soil properties. The $Ca(H_2PO_4)_2$-S was found to be higher in all the soil types while the water soluble S content was found to be low. Black soils witnessed higher content of extractable S fractions compared to alluvial and red soils. Different magnitude relationships were observed between soil properties and extractable S content and among the different fractions of extractable S showed. Based on the results it was concluded that the soil properties could alter the different fractions of S in soils and their availability for crop nutrition.

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