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Studies on relationships of soil acidities and sulphur fractions with soil properties of continuously fertilized acid Alfisol of western Himalayas

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Abstract

The present investigation was undertaken in the ongoing long-term fertilizer experiment initiated during 1972 at experimental farm of Department of Soil Science, Himachal Pradesh Agricultural University, Palampur (India). Different forms of soil acidity (total acidity, total potential acidity, pH dependent acidity, extractable acidity, exchangeable acidity and non-exchangeable acidity) significantly and positively correlated with organic carbon, available NPK, CEC and micronutrient cations, whereas negatively correlated with soil pH. Besides, different fractions of sulphur *i.e.* available S, water soluble S, heat soluble S, organic S and total S significantly and positively correlated with organic carbon, available NPK and CEC, whereas non significant negatively correlated with soil pH

Keywords: Amendments, correlation, fertilizers, forms of soil acidity, lime, long-term effect, yield

Introduction

In India, intensive agriculture during the past three decades, involving exhaustive highvielding varieties of maize and wheat has led to heavy withdrawal of nutrients from the soil. Furthermore, imbalanced use of chemical fertilizers by farmers has deteriorated soil health. The widely practiced maize-wheat system in India is one such instance, where sustainability is under threat. Imbalanced use of fertilizers has increased the problem of soil acidity and also raised the problem of sulphur deficiency. Soil acidity is as a major constraint to crop production throughout the world (Sumner and Noble 2003)^[32]. Sulphur is the fourth most important nutrient element after nitrogen, phosphorus and potassium for crop production. It is gaining considerable importance in the context of Indian agriculture, particularly when there is more and more use of non-sulphur containing fertilizers as well as organic manures (Raina and Tanawade 2005) [25]. In India, acid soils constitute nearly one-third of the area under cultivation. Acid soils are widely distributed in Himalayan regions, Eastern, North-Eastern and in Southern states under varying climatic and environmental conditions. Addition of acid forming fertilizers, intensive cultivation and heavy irrigation also result in the development of acidity in these soils. Hence, these soils are poor in basic cations (Dhananjaya and Ananthanarayana 2010)^[9]. The distribution of cationic micronutrients especially Fe²⁺, Mn^{2+,} Cu2+ and Zn2+ in acid soils of India is sporadic. The different forms of acidity showed significant positive correlations with organic carbon and but negative correlations with soil pH (Pati and Mukhopadhyay 2010)^[23]. In Himachal Pradesh, acid soils having pH less than 6.5 occupy around 1.78 m ha (Sharma et al. 2006).

Knowledge of different forms of acidity is the first step in understanding the nature of acid soils for their better management (Dolui and Mehta 2001). Also the knowledge about different forms of sulphur and their relationship with some important soil characteristics decide the sulphur supplying power of soil by influencing its release and dynamics. However, knowledge of different forms of sulphur in soil along with their distribution in the zone of penetration is of much relevance in assessing the long-term availability of nutrients and in formulating strong fertilizer recommendations. Therefore, the present study was undertaken to investigate the effect of continuous cropping with different fertilizers and amendments on relationship of different forms of soil acidity and sulphur with different soil properties.

Materials and Methods

The study to investigate the effect of continuous cropping with different fertilizers and amendments on relationship of different forms of soil acidity and sulphur with different soil properties was carried out in an ongoing fertilizer experiment started during 1972 at experimental farm of Department of Soil Science, CSK Himachal Pradesh Agricultural University, Palampur, located at $31^{\circ}6'$ N latitude and $76^{\circ}3'$ E longitude, 1290 meters above mean sea level. Average annual rainfall of the study area was 2500-3000 mm with wet temperate climate. The mean maximum temperature remains about 30°C during the hottest months of May to June. December to February were the coldest months with minimum temperature of about 3 °C. At the start of the experiment the soil of the study area was silty loam in texture and as per the taxonomic system of soil classification (Soil Survey Staff 1975) classified as Typic Hapludalf. Some important physicochemical characteristics of the surface soil (0 - 0.15 m) at the initiation of the experiment are given in table a. Initially, the experiment comprised of 10 treatments. The 11th treatment viz., 100% NPK (-S) was introduced in kharif 1981. The experiment has been laid out in randomized block design. The detail of treatments has been given in table b. Due to build up in available P, the treatments have been modified slightly from kharif 2011, the optimal and super optimal dose of P has been reduced by 50 per cent and in case of sub optimal dose of NPK (T₁), FYM @ 5t ha⁻¹ has also been included.

The recommended dose of N, P2O5 and K2O for wheat was 120, 60 and 30 kg ha⁻¹ and for maize 120, 60 and 40 kg ha⁻¹, respectively. Half dose of N and full dose of P and K were applied at the time of sowing in both the crops. The remaining half nitrogen was top dressed in two equal splits at maximum tillering and flowering stage of wheat and knee high and pretasseling stages in maize crop, respectively. The sources of N, P and K were urea, single super phosphate and muriate of potash, respectively. In NPK (-S) treatment, P was applied through diammonium phosphate (DAP) to assess the effect of 'S' free high analysis P fertilizer in crop production. Zinc was applied in T₅ as zinc sulphate @ 25 kg ha^{-1} every year to both the crops till rabi 2010-11. FYM was applied @ 10 t ha-1 on fresh weight basis to maize crop only till rabi 2010-11 and from kharif 2011 FYM was applied @5t ha-1on dry weight basis. The FYM applied contained 60 per cent moisture and its average nutrient content on dry weight basis was 1.01, 0.26 and 0.40 per cent of N, P and K, respectively. Lime was added in T₁₀ @ 900 kg ha⁻¹ as marketable lime (CaCO₃) passed through 100 mesh sieve to maize crop only. Lime application continued till the soil pH was raised to about 6.5. Lime application in the subsequent years was restored only when the soil pH declined to about 6.3. In case of wheat, irrigations were given at critical growth stages. In case of maize crop, one pre-sowing irrigation was given. Thereafter, the crop met its water requirement through rainfall. Chemical

weed control measures were followed in both the crops except in T_4 (100 % NPK + hand weeding) where weeds were removed manually.

Soil samples collected from a depth of 0-0.15, 0.15-0.30 and 0.30-0.45 m were used for determination of various chemical parameters. The processed soil samples were analyzed for soil pH, organic carbon, cation exchange capacity, available nitrogen, phosphorus, and potassium. Combined glasscalomel electrode was used to determine the pH (Jackson 1973) of aqueous suspensions (1:2.5 soil/solution ratio), organic carbon was measured with the wet digestion method (Walkley and Black 1934), cation exchange capacity (Jackson 1973), available N was measured with the alkaline permanganate method (Subbiah and Asija 1956), available P was found using the bicarbonate extraction method (Olsen et al. 1954), available K was determined by the neutral normal ammonium acetate extraction method (Black 1965) and DTPA extractable micronutrient cations (Fe, Mn, Zn and Cu) were determined by Atomic Absorption Spectrophotometer (AAS) (Lindsay and Norvell 1978). The soil samples were also analyzed for forms of soil acidity viz., exchangeable acidity, total potential acidity, pH dependent acidity, extractable acidity, non-exchangeable acidity and total soil acidity. Exchangeable acidity was determined by the method as outlined by Mclean (1965). Total potential acidity was measured by using 0.5 N BaCl₂ + triethanolamine (pH - 8.0 -8.2) reagent as extractant (Baruah and Barthakar 1998). The pH dependent acidity was determined by subtracting the exchangeable acidity from total potential acidity. For extractable acidity, soil samples were extracted with NH₄OAc solution (pH 4.8) and the values were determined by colorimetric method (Baurah and Barthakar 1998). Nonexchangeable acidity was calculated by subtracting the value of exchangeable acidity from extractable acidity. Total soil acidity was estimated by extracting the soil with freshly prepared N NaOAc solution (pH 8.2) as per extraction procedure given by Kappen (1934). Different forms of sulphur were determined in the soil samples collected after the harvest of maize (kharif, 2014) following standard procedures. Total sulphur was determined by the method as outlined by Johnson and Nishita (1952). Water soluble sulphur was estimated turbidimetrically using de-ionized water as extracting solution (Chesnin and Yien 1950). Heat soluble sulphur was determined turbidimetrically (Williams and Steinbergs 1959). The sulphate sulphur in soil extract was determined colorimetrically by developing BaSO4 turbidity in the presence of sodium acetate-acetic acid buffer (Chesnin and Yien 1950). Organic sulphur was calculated by subtraction of sulphate sulphur from total sulphur. The data generated from field and laboratory analyses were subjected to statistical analysis using the technique of analysis of variance for randomized block design for the interpretation of results as described by Gomez and Gomez (1984).

 Table a:
 Some initial (1972) physico-chemical characteristics of the surface soil

Parameter	Value
Bulk density (Mg m ⁻³)	1.31
Mechanical Separates (%)	
Sand	29
Silt	47
Clay	24
Textural class	Silty loam
Taxonomic classification	Typic Hapludalf
Organic carbon (g kg ⁻¹)	7.9
CEC [cmol (P^+) kg ⁻¹]	12.1

pH (Soil reaction)	5.8 (acidic)
Available nutrients (kg ha ⁻¹)	
Ν	736.0
Р	12.0
Κ	194.2
DTPA- Extractable micronutrient cations (mg kg ⁻¹)	
Fe^{2+} Zn^{2+}	26
Zn^{2+}	1.9
${ m Mn}^{2_+} { m Cu}^{2_+}$	24.3
Cu^{2+}	0.4

Table b: The treatments are as follows

T ₁	50% NPK (sub optimal)
T_2	100% NPK
T3	150% NPK (Super optimal)
T ₄	100% NPK + Hand Weeding (HW)
T ₅	100% NPK + Zn @ 25 kg ha ⁻¹ through ZnSO ₄
T ₆	100% NP
T7	100% N
T ₈	100% NPK + FYM @ 10 t ha ⁻¹ (to maize crop only)
T9	100% NPK (-S), P through DAP
T ₁₀	100% NPK + lime @ 900 kg ha ⁻¹
T ₁₁	Control

Results and Discussion

Relationship of sulphur fractions with pH, OC, available NPK and CEC in soil

Relationship of sulphur fractions with pH

A close look on the data (Table 1) depicted that a non significant relationship between pH and S fractions was obtained. The maximum value of correlation coefficient between S fractions and pH was obtained in between organic S and pH (r = 0.189) followed by total S and heat soluble S. While, with available S and water soluble S, it was negative with correlation values, -0.125 and -0.061, respectively. Such negative correlations might be due to abundance of Fe and Al oxides in soils that caused more adsorption of S from soil solution. Lower solution pH. which controls the polarity and surface density of adsorption plane, like Fe and Al oxides, enhances the S adsorption. (Basumatari et al. 2010). Negative correlation between water soluble S and pH was also reported by (Basumatari et al. 2010). As pH increases sulphur sorption decreases which in turn supplements the water soluble S in soil solution. Das et al. (2011) also recorded a negative correlation between water soluble S and pH and concluded that soil pH was responsible for regulating concentration of S in soil solution. Positive but not significant relation between pH and S fractions were also reported by Sharma et al. (2014).

 Table 1: Coefficients of correlation (r) between sulphur fractions and pH and organic carbon in surface soil

Sulphur fractions	pН	OC
Available S	-0.125	0.579**
Water soluble S	-0.061	0.690**
Heat soluble S	0.124	0.638**
Organic S	0.189	0.647**
Total S	0.144	0.654**

**Significant at 1 % level of significance

Relationship of sulphur fractions with soil organic carbon: S fractions were positively and significantly correlated with soil organic carbon (Table 1). The highest value of correlation was recorded in between water soluble S and SOC (r = 0.690) followed by total S, organic S, heat soluble S and available S with correlation values 0.654, 0.647, 0.630 and 0.579, respectively. The significant and positive correlation of total S with SOC has also reported earlier by (Sharma and Jaggi 2001; Borkotoki and Das 2008; Basumatari et al. 2010). Correlation studies indicated a highly significant positive correlation of organic S with organic carbon (r=0.647), this may be due to the fact that organic carbon is the main source of organic S; therefore positive significant correlations are expected. Basumatary et al. (2008) and Borkotoki and Das (2008) had also made similar observations on soils of Assam. Jat and Yadav (2006) also reported same results. The observed significant positive correlation of available S with SOC suggests that sulphur supplying power of these soils was largely dependent upon this parameter (Basumatari et al. 2010). There existed a positive and significant correlation between water soluble S and organic S and these results corroborate the observations of Basumatari et al. (2010) and Das et al. (2011).

Relationship of sulphur fractions with available nitrogen

Available N at surface layer (0 - 0.15 m) was positively and significantly correlated with all the sulphur fractions (Table 2). Heat soluble sulphur (HSS) (r = 0.617) showed the highest correlation value with available N followed by total S (r = 0.588), available S (r = 0.578), organic S (r = 0.572) and WSS (r = 0.563) in surface soil layer (0 - 0.15 m). The positive and significant correlation between S fractions and available N was typical because S and N are the integral constituents of proteins in organic matter (Basumatari *et al.* 2010).

In subsurface layer (0.15 - 0.30 m), the highest correlation of available N was found with HSS (r=0.536) followed by WSS and organic S with correlation values 0.514 and 0.347, respectively. The correlation was positive but not significant in case of available S and total S with values 0.332 and 0.332, respectively. In soil layer 0.30 - 0.45 m, there was a positive but non significant correlation with all the S fractions and available N. The highest correlation was obtained with organic S followed by total S, HSS, WSS and available sulphur with values 0.253, 0.252, 0.245, 0.227 and 0.168, respectively.

Table 2 Coefficients of correlation (r) between sulphur fractions CEC and available NPK in soil

Sulphur fractions	Ν	Р	K	CEC			
0-0.15 m							
Available S	0.578**	0.740**	0.574**	0.514**			
Water soluble S	0.563**	0.723**	0.607**	0.583**			
Heat soluble S	0.617**	0.574**	0.437*	0.655**			
Organic S	0.572**	0.682**	0.526**	0.717**			
Total S	0.588**	0.709**	0.548**	0.704**			

	0.15-0.30) m			
Available S	0.332	0.820**	0.618**	0.604**	
Water soluble S	0.514**	0.731**	0.518**	0.779**	
Heat soluble S	0.536**	0.632**	0.502**	0.671**	
Organic S	0.347*	0.793**	0.526**	0.677**	
Total S	0.332	0.790**	0.528**	0.661**	
	0.30-0.45	5 m			
Available S	0.168	0.833**	0.569**	0.673**	
Water soluble S	0.227	0.793**	0.388*	0.707**	
Heat soluble S	0.245	0.496**	0.608**	0.717**	
Organic S	0.253	0.694**	0.475**	0.728**	
Total S	0.252	0.705**	0.482**	0.730**	

**Significant at 1 % level of significance

*Significant at 5 % level of significance

Relationship of sulphur fractions with available phosphorus A significant and positive relationship between available P and sulphur fractions in all the layers of soil up to 0.30 - 0.45m soil layer (Table 2). The highest association between S fractions and available P was obtained with available S at all the three depths with values 0.740, 0.820 and 0.833 in 0 -0.15, 0.15 - 0.30 and 0.30 - 0.45 m depth, respectively. Likewise, the lowest association was obtained with HSS at all the three layers with values 0.574, 0.632 and 0.496 in 0 - 0.15, 0.15 - 0.30 and 0.30 - 0.45 m depth, respectively. In surface layer, the correlation values can be arranged in descending order with the highest value in available S followed by WSS, total S, organic S and then HSS (Table 4.24). Similar trend was followed in subsurface layer of depth 0.30 - 0.45 m. In 0.15 - 0.30 m soil layer, the correlation values can be arranged in descending order with the highest value in available S followed by organic S, total S, WSS and then HSS.

Relationship of sulphur fractions with available potassium

Available K was also positively and significantly correlated with all the fractions of sulphur similar to available N and available P (Table 2). Among the various sulphur fractions, the highest correlation (r = 0.607) of available K was found with WSS followed by available S (r = 0.574), total S (r =0.548), organic S (r = 0.526) and HSS (r = 0.437) in surface layer. Likewise, in subsurface layer (0.15 - 0.30 m), the available K exhibited positive and significant relationship with different sulphur fractions. The maximum correlation of available K was found with available S (r = 0.618) followed by total S, organic S, WSS and HSS and values of coefficient of correlation (r) were 0.528, 0.526, 0.518 and 0.502, respectively. In lower depth 0.30 - 0.45 m, there also existed a positive and significant correlation among S fractions and available K. At this depth the highly associated fraction with available K was HSS (r=0.608) followed by available S (r=0.569), total S (r=0.482), organic S (r=0.475) and then by WSS (r=0.388).

Relationship of sulphur fractions with CEC

S fractions were positively and significantly correlated with CEC in all the three layers of soil (Table 2). In surface layer (0 - 0.15 m), the highest value of correlation was obtained in between organic S and CEC (r = 717) which was followed by total S, heat soluble S, water soluble S and available S with correlation values 0.704, 0.655, 0.583 and 0.514, respectively. In subsurface layer (0.15 - 0.30 m) the highest correlation was obtained with water soluble S (r = 0.779) and the lowest with available S (r=0.604). In soil layer 0.30 - 0.45 m, total S was the fraction which was highly correlated with CEC (r = 0.730) followed by organic S (r=0.728), heat soluble S (r=0.717), water soluble S (r=0.707) and available sulphur (r=0.673). Further, a close look on the data revealed that the lowest value of correlation between S fractions and CEC was obtained with available S in all the three soil layers. Basumatari et al. (2010), Das et al. (2011), Patel et al. (2011) and Sharma et al. (2014a), also reported the positive and significant relationship in between CEC and S fractions. In case of water soluble S Das et al. 2011 reported that in many cases the relationship was found to be significant.

Parameters	pН	OC	Ν	Р	K	CEC
Exchangeable acidity	-0.749**	0.426**	0.645**	0.458**	0.364**	0.173
Total potential acidity	-0.681**	0.531**	0.603**	0.527**	0.524**	0.281**
pH dependent acidity	-0.596**	0.509**	0.533**	0.495**	0.519**	0.285**
Extractable acidity	-0.806**	0.386**	0.623**	0.408**	0.314**	0.098
Non exchangeable acidity	-0.819**	0.296**	0.539**	0.299**	0.214*	-0.017

**Significant at 1 % level of significance *Significant at 5 % level of significance

Relationship of different forms of soil acidity pH, OC, available NPK and micronutrient cations in soil Relationship of forms of soil acidity with pH

A significant and negative relationship between pH and different forms of soil acidity was obtained (Table 3). The maximum value of correlation coefficient between soil acidity forms and pH was obtained in between pH dependent acidity and pH ($r = -0.596^{**}$), whereas, minimum value was found with non-exchangeable acidity ($r = -0.819^{**}$). Dolui *et al.*

(2010) and Pati and Mukhopadhyay (2010) ^[23] in their correlation studies also reported that the pH had a significant negative correlation with all types of acidity. It was observed that the pH had a significant negative correlation with different forms of soil acidity, suggesting that, these forms of acidity were responsible for lowering the pH of the soils. The soil pH negatively correlated significantly (p < 0.05 and 0.01) with exchangeable acidity was also recorded by Nwite *et al.* (2016).

Relationship of forms of soil acidity with soil organic carbon Soil acidity forms were positively and significantly correlated with soil organic carbon (Table 3). The highest value of correlation was recorded in between total potential acidity and SOC (r = 0.531) followed by pH dependent acidity, exchangeable acidity, extractable acidity and non exchangeable acidity with correlation values 0.509, 0.426, 0.386 and 0.296, respectively. The significant and positive correlation of different forms of soil acidity with SOC has also reported earlier by Bandyopadhyay and Chattopadhyay (1996) in their experiment conducted at Alfisols and Inceptisols of West Bengal. Similar results were also reported by Dolui et al. (2010) and Patton et al. (2007). Organic C had significantly positive correlations with pH-dependent and total potential acidities. Soil organic matter possesses number of functional groups containing H⁺ ions which can contribute to different forms of acidities depending upon their magnitude (Laxminarayana 2010).

Relationship of forms of soil acidity with available nitrogen

Available N was positively and significantly correlated with all forms of soil acidity (Table 3). Exchangeable acidity (r = 0.645) showed the highest correlation value with available N followed by extractable acidity (r = 0.623), total potential acidity (r = 0.603), non- exchangeable acidity (r = 0.539) and pH dependent acidity (r = 0.533).

Relationship of forms of soil acidity with available phosphorus

The significant and positive correlation between forms of soil acidity and available P was obtained with total potential acidity (r = 0.527) (Table 3). Likewise, the lowest association was obtained with non- exchangeable acidity with value 0.299. The correlation values can be arranged in descending order with the highest value in total potential acidity followed by pH dependent acidity, exchangeable acidity, extractable acidity and then non-exchangeable acidity.

Relationship of forms of soil acidity with available potassium

Available K was also positively and significantly correlated with all forms of soil acidity similar to available N and available P (Table 3). The highest correlation (r = 0.524) of available K was found with total potential acidity followed by pH dependent acidity (r = 0.519), exchangeable acidity (r = 0.364), extractable acidity (r = 0.314) and non- exchangeable acidity (r = 0.214).

Relationship of forms of soil acidity with CEC

Total potential acidity and pH dependent acidity were positively and significantly correlated with CEC, whereas, non- exchangeable acidity was negatively correlated with CEC (r = 0.017) (Table 3). The highest value of correlation was obtained in between pH dependent acidity and CEC (r = 0.285) which was followed by total potential acidity with correlation values 0.281. Dolui and Mehta (2011) in their experiment also reported that organic carbon did not show significant correlation with exchange acidity, whereas, positive correlation was recorded with total potential acidity. Cation exchange capacity had significant positive correlation with pH-dependent acidity (Patton *et al.* 2007).

Relationship of forms of soil acidity with micronutrient cations (Fe, Cu, Mn and Zn)

different forms of soil acidity and micronutrient cations (Table 4). Significant positive correlation of Mn⁺² with total potential acidity (0.845), pH-dependent acidity (0.796), exchangeable acidity (0.729), extractable acidity (0.721), and non- exchangeable acidity (0.646) was observed (Table). The total potential acidity and pH-dependent acidity had the major contribution to DTPA extractable-Mn²⁺. Significant positive correlations of DTPA extractable-Cu²⁺ with total potential acidity (0.660), pH-dependent acidity (0.639), exchangeable acidity (0.510), extractable acidity (0.507), and nonexchangeable acidity (0.457) were observed (Table 1). Significant positive correlations of the DTPA extractable Zn²⁺ with total potential acidity (0.476), pH-dependent acidity (0.452), extractable acidity (0.406), exchangeable acidity (0.399) and non- exchangeable acidity (0.381) were observed (Table 1). Total potential acidity had the major contribution to the variation of the DTPA extractable Zn²⁺. Significant positive correlations of the DTPA extractable Fe²⁺ with total potential acidity (0.891), pH-dependent acidity (0.871), exchangeable acidity (0.657), extractable acidity (0.643) and non- exchangeable acidity (0.568) were observed. Pati and Mukhopadhyay (2010)^[23] also reported the similar results.

Conclusion

It is therefore concluded that the different forms of soil acidity and soil sulphur are significantly positively correlated with different soil properties except pH. Soil acidities forms are significantly negatively correlated with pH, whereas, in case of sulphur fractions the relationship with pH is nonsignificant. The different forms of acidity governed the distribution of the extractable –micronutrients in soils and positively correlated. Exchangeable acidity has non significant relationship with cation exchange capacity and non exchangeable acidity has negative correlation with cation exchange capacity.

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