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Sulfur Distribution and Availability in Alkaline Subtropical Soils Affected by Organic Amendments

Kouser Majeed Malik[1] · Khalid Saifullah Khan[1] · Mohammad Saleem Akhtar[1] · Zammurad Iqbal Ahmed[2]

Abstract

Sulfur availability depends upon the distribution of S forms in soil, interaction among them, and soil microbial properties. Organic amendments play a vital role in maintaining adequate S reserves in soil. However, two important questions in this regard are (i) the effect of microbial activity on sulfur oxidation in soil, and (ii) the relationship of sulfur fractions to available S pool in soil. The present experiment was designed to assess the effect of organic amendments on sulfur fractions, availability, and microbial properties in soil. Two alkaline subtropical soils belonging to Missa (*silt loam Typic Ustochrept*) and Kahuta (*sandy loam Udic Haplustalf*) soil series were amended with organic amendments, viz. farmyard manure (FYM), poultry litter (PL), and sugarcane filter cake (SF), at 1% w/w and incubated at 25 °C for 56 days maintaining soil moisture at 50% WHC. CO_2-C evolution rate, the $\sum CO_2$-C, and also the $\sum CO_2$-C to microbial biomass C differed with the soils and were higher in poultry litter–amended soils. Dissolved organic C (DOC), microbial biomass C (MBC), and dehydrogenase activity (DHA) were also higher in poultry litter while the microbial biomass S (MBS) and arylsulfatase activity (ASA) were higher in the sugarcane filter cake–amended soils. Organic amendments significantly increased plant-available SO_4^{2-} and enhanced C-bonded S, and inorganic S fractions in the following order: sugarcane filter cake > poultry litter > farmyard manure. Organic amendments significantly improved microbial activity and S availability in soil depending upon their labile organic C, organic S, and available S contents.

Keywords Microbial biomass · Enzyme activity · Dehydrogenase activity · Organic amendments · Sulfur fractions · Alkaline calcareous soils

1 Introduction

Sulfur availability to crop plants depends upon different S forms present in soil and interaction among them (Parakhia et al. 2016). Organic C content, type of fertilizers used especially the organic fertilizers, and soil pH are the main factors influencing sulfur distribution to soil and its release into plant-available form (Gao et al. 2017). Conversion of organic S into inorganic sulfate and the reverse processes play a vital role in the biogeochemical cycling of S and its bioavailability (Blum et al. 2013; Xu et al. 2016). Studies have shown that organic amendments help to maintain adequate soil S reserves, particularly in organic S forms such as the C-bonded S and ester sulfate S (Wyngaard and Cabrera 2015; Xu et al. 2016). Both of these organic S fractions may be converted into inorganic sulfate through microbial-mediated processes (Solomon et al. 2011). Ester S has a higher mineralization rate indicating more bioavailability of S from this source (Yang et al. 2007). Thus, the relative proportion of C-bonded S and ester sulfate S in soil's organic S pool is important to assess S mineralization and its availability to crop plants.

Soil microbial biomass is the key factor regulating sulfur transformations in soil (Yang et al. 2007). The labile nature of microbial biomass S makes it an important component of the soil sulfur cycle. Although microbial biomass S constitutes less than 5% of the total soil sulfur, it performs a key function

✉ Khalid Saifullah Khan
khalidsaifullah@uaar.edu.pk

Kouser Majeed Malik
kousermajeed9@gmail.com

Mohammad Saleem Akhtar
msakhtar@uaar.edu.pk

Zammurad Iqbal Ahmed
azammurad@hotmail.com

[1] Institute of Soil Science, Pir Mehr Ali Shah Arid Agriculture University, Rawalpindi 46300, Pakistan

[2] Department of Agronomy, Pir Mehr Ali Shah Arid Agriculture University, Rawalpindi 46300, Pakistan

in sulfur nutrition of crop plants due to rapid mineralization. Fluxes in microbial biomass S strongly correlate with the concentration of inorganic SO_4^{2-} in soils (Scherer 2009). The addition of organic amendments may contribute to improvement of the nutrient status and soil microbial function in arid and semi-arid calcareous soils with very low OM content (Karimi et al. 2020; Khadem and Raiesi 2017). Organic amendments stimulate the size and activity of soil microbial biomass (St-Pierre and Wright 2017; Suleman et al. 2019) and the increased contents were reported with the addition of farmyard manure (Yang et al. 2007), glucose-C (Suleman et al. 2019), and biochar (Karimi et al. 2020). Lalande et al. (2003) observed a 55% rise in microbial biomass C and a 30% increase in enzyme activity as a result of manure and sludge applications. Lupwayi et al. (2005) found twofold to threefold increases in microbial biomass C with hog manure and cattle manure application, respectively. Soil respiration as a measure of CO_2-C released from the soil, microbial biomass N, and microbial biomass S also increased in response to the addition of different organic amendments (Howlader et al. 2008). A good portion of the increased soil respiration is likely due to the microbial use of the added organic C as well as priming of the native soil organic matter (Lajtha et al. 2017).

Concerns regarding changes in sulfur fractions in soil prompted studies on sulfur speciation under different management systems (Yang et al. 2007; Blum et al. 2013). Conflicting results were reported regarding organic amendments effect on S transformation and distribution in soil (Balik et al. 2009; Xu et al. 2016; Yang et al. 2007). Two important questions in this regard were as follows: (i) how do organic amendments affect microbial properties and sulfur fractions in soils, and (ii) what is the relationship of sulfur fractions to available S pool in soil? Based on the above discussion, it is hypothesized that "organic amendments stimulate microbial activity and improve sulfur availability in alkaline soils." The information is in particular less documented for the alkaline soils of arid and semi-arid tropical/subtropical regions having low (< 1%) organic matter contents (Malik et al. 2013). These soils are characterized by their low fertility status and require heavy inputs of nutrients for successful crop production (Karimi et al. 2020; Wani et al. 2016). In view of the above, the present experiment was designed with the objective to assess the influence of organic amendments on sulfur distribution, availability, and microbial properties of organic matter–deficient alkaline subtropical soils.

2 Materials and Methods

2.1 Collection of Soils and Organic Amendments

Two sulfur-deficient soils belonging to the series Missa (*inceptisol*, *Typic Ustochrepts* 33° 22′ 09.1″ N 72° 29′ 08.5″

E) and Kahuta (*alfisol*, *Udic Haplustalfs* 33° 04′ 07.1″ N 73° 00′ 30.7″ E) were sampled from the agricultural fields located at University Research Farm (URF), Koont, and farmer's field, Fateh Jang, in Northern Punjab, Pakistan. Composite soil samples weighing approximately 50 kg each were taken from the field surface at 0–15-cm depth, transported to the laboratory, sieved (< 2 mm) in moist condition, homogenized, and kept frozen at − 15 °C until the incubation started. Subsamples comprising 500 g of each soil were air-dried, crushed, sieved (< 2 mm), and subjected to analysis for basic physical and chemical characteristics, viz. moisture content, particle size distribution, water-holding capacity, pH, electrical conductivity (EC), calcium carbonate ($CaCO_3$), cation exchange capacity (CEC), total organic carbon, total nitrogen, Olsen phosphorous, extractable potassium, available sulfate (SO_4^{2-}), and AB-DTPA extractable iron, copper, manganese, and zinc. Among the organic amendments used in the experiments, farmyard manure was arranged from the cattle manure heap in a farmer's field at Taxila; poultry litter was taken from the Poultry Research Institute, Rawalpindi; and the sugarcane filter cake was brought from the industrial dump of the Koh-e-Noor Sugar Mills, Jauharabad. The organic amendments were air-dried, ground, homogenized, and analyzed for their chemical composition.

2.2 Incubation Experiment

The frozen soil samples were kept at room temperature for 1 day, with moisture contents adjusted to 50% of their water-holding capacity, and pre-incubated (07 days) at 25 °C prior to organic amendments application. The soils were transferred to incubation jars separately at the rate of 600 g per jar on a dry weight basis and the following treatments were applied: (i) control, (ii) farmyard manure (FYM), (iii) poultry litter (PL), and (iv) sugarcane filter cake (SF). Each organic amendment was added to the soil on a 1% w/w basis; i.e., 6 g of the amendment was applied to each jar containing 600 g of soil (Rasul et al. 2008). The treatments were replicated three times following the completely randomized design (CRD). The lids of the incubation jars were closed airtight and the incubation was carried out in dark for a period of 56 days in an incubator set at 25 °C. The CO_2 released was captured in 1 M NaOH solution placed in each incubation jar in small glass vials and estimated by back-titration with 1 M HCl periodically after 1, 2, 3, 5, 7, 10, and 14 days and afterwards weekly. Fifty-gram soil on dry weight basis was taken from each incubation jar at days 0, 14, 28, 42, and 56 and analyzed for dissolved organic C (DOC), microbial biomass C (MBC), microbial biomass S (MBS), arylsulfatase activity (ASA), dehydrogenase activity (DHA), and available sulfate. Soil samples collected on days 0, 28, and 56 were further analyzed for sulfur fractions.

2.3 Analytical Methods

Particle size analysis was performed according to Gee and Bauder (1979) using a standard hydrometer after pretreatment of soil samples with a 1% solution of sodium hexametaphosphate. Soil pH and EC were measured in a saturated soil paste using a calibrated pH meter (HANNA-212) and a conductivity meter (HANNA HI-8033), respectively. Calcium carbonate content was determined by acid neutralization method (Rayan et al. 2001). Cation exchange capacity was estimated following the procedure given by Anderson and Ingram (1993). Total organic C was determined after dichromate digestion by modified Mebius procedure (Nelson and Sommers 1982). Total N was determined by using the Kjeldahl Distillation Apparatus (Bremner and Mulvaney 1982). The Olsen P was measured spectrophotometrically using 0.5 M $NaHCO_3$ (pH 8.5) as an extractant (Anderson and Ingram 1993). Soil microbial biomass C and microbial biomass S contents were measured by chloroform fumigation-extraction methods (Khan et al. 2009; Vance et al. 1987). Fumigated and non-fumigated samples of 10 g soil were extracted with 40 ml 0.5 M K_2SO_4 for MBC and 50 mM NH_4NO_3 for MBS by horizontal shaking at 200 revolutions per minute for 30 min and filtered. Microbial biomass C was calculated by measuring organic carbon in the extracts as CO_2 by infrared absorption after combustion at 760 °C using a Shimadzu automatic TOC analyzer and calculated by the following formula: Microbial biomass C = E_C/k_{EC}, where E_C = (organic C extracted from the fumigated soil) − (organic C extracted from non-fumigated soil), and k_{EC} = 0.45. Microbial biomass S was calculated by the following formula: Microbial biomass S = E_S/k_{ES}, whereas E_S = (total S extracted from the fumigated soil) − (total S extracted from the non-fumigated soil), and k_{ES} = 0.35.

Arylsulfatase activity was measured by the procedure described by Tabatabai and Bremner (1970). In 1-g soil, 4 ml of 0.5 M acetate buffer (pH 5.8), 0.25 ml toluene, and 1 ml of 50 mM potassium p-nitrophenylsulfate solution were added, and the contents were incubated at 37 °C for an hour. 1 ml of 0.5 M $CaCl_2$ and 4 ml of 0.5 M NaOH were added and filtered. The yellow color developed of p-nitrophenol was measured by spectrophotometrically (Cecil-2000) at 400-nm wavelength. Dehydrogenase activity was measured via a procedure proposed by Casida et al. (1964). Twenty-gram soil and 0.2 g of $CaCO_3$ were mixed and the contents were equally divided into three test tubes. One milliliter of 3% triphenyl tetrazolium chloride (TTC) solution and 2.5 ml of de-ionized water were added and incubated for 24 h at 37 °C. Samples were shaken after adding 10 ml of methanol and then filtered. The intensity of red color was measured spectrometrically at 485-nm wavelength. The TPF standard calibration curve was used as a reference to quantify the TPF produced.

The sequential extraction procedure proposed by Morche (2008) was followed to extract inorganic sulfur fractions in soils. For 1.0 g soil, 10 ml of de-ionized water (1:10 w/v) was added; contents were shacked for 1 h on a reciprocating shaker and centrifuged at 10000 rpm for 10 min. The clear supernatant was collected for measuring water-soluble plant-available sulfur. The residual soil in the centrifuge tube was added with 0.032 M NaH_2PO_4 solution (1:10 w/v); the contents were agitated for 1 h on a reciprocating shaker and centrifuged at 10000 rpm. The aliquot collected was used to determine weakly adsorbed sulfur. Twenty milliliters of 1 M HCl (1:20 w/v) was added to residual soil, contents were agitated for 1 h and centrifuged at 10000 rpm for 10 min, and the supernatant was collected to determine occluded or acid-soluble S. Sulfur contents in the sequential extracts were determined by measuring the turbidity (Verma et al. 1977). Organic sulfur fractions in soil including ester sulfate S and C-bonded S were analyzed following the procedure proposed by Johnson and Nishita (1952). HI-reducible sulfur was measured by treating soil with a mixture of hydriodic acid, hypophosphoric acid, and formic acid. Resulting H_2S was absorbed in NaOAc-$Zn(OAc)_2$ solution and the sulfide was measured spectrophotometrically at 670-nm wavelength after methylene blue color development by the addition of N,N-dimethyl-p-phenylene diamine sulfate and ferric ammonium sulfate. Ester-bonded S was calculated from the difference between HI-reducible S and inorganic S. Carbon-bonded S was calculated by subtracting HI-reducible S from the total S contents of the soil. Available SO_4^{2-} in the soil was measured by extracting 5 g of air-dried soil with 25 ml of 0.01 M $CaCl_2$ solution and determining the SO_4^{2-} contents in extracts by turbidimetric method (Verma et al. 1977).

2.4 Statistical Analyses

The tabulated data represented are arithmetic means of three replicates on an oven dry weight basis (105 °C, 24 h). The significance of soil, organic amendments (OA), and days of incubation (DOI) were tested using a three-way analysis of variance. Least significant difference (LSD) test was applied to evaluate differences among the treatment means at 0.05% level of significance. All the statistical analyses were performed using Statistix 8.1 software. The relationship between available S and other sulfur fractions was observed by calculating the correlation coefficients.

3 Results

3.1 Characteristics of Soils and Organic Amendments

The Missa soil used in this experiment was silt loam and the Kahuta soil was sandy loam in texture. Both the soils were

non-saline, moderately alkaline, and calcareous with pH levels of 7.5 and 7.2 and $CaCO_3$ contents of 13.4 and 5.3%, respectively (Table 1). The CEC of Missa was relatively higher than that of the Kahuta soil. Both the soils were deficient in plant-available sulfur (< 10 μg g^{-1} soil) and low in organic matter with total organic C contents less than 0.5%. Total nitrogen, available phosphorous, and extractable potassium contents of the soils were low. AB-DTPA extractable iron contents were also low in both the soils whereas the extractable manganese, copper, and zinc were above the permissible limit described by Soltanpour (1985). The three organic amendments, i.e., farmyard manure, poultry litter, and sugarcane filter cake, differed in their chemical composition (Table 2). Moisture content was higher in PL as compared with that in FYM and SF. Total organic carbon, total nitrogen, total phosphorous, and total zinc contents were higher in PL while the total iron and total manganese contents were higher in FYM in comparison with the other two amendments. The concentrations of toxic metal (As, Cd, Cr Hg, Ni, Pb) in all the organic amendments were within the range of permissible limits described by Bloem et al. (2017). Total S was high in SF, followed by PL and FYM. The ratio of total organic C to total S was lowest in SF, whereas the ratio of total organic C to total N and total organic C to total P was lowest in the PL.

Table 1 Physical and chemical properties of soils

Soil properties	Missa soil	Kahuta soil
Sand (%)	37.5 ± 0.20	47.5 ± 2.30
Silt (%)	54.0 ± 1.72	34.0 ± 2.06
Clay (%)	8.5 ± 1.15	18.5 ± 1.93
Textural class	Silt loam	Sandy loam
Water-holding capacity (%)	36 ± 2.78	30 ± 2.53
Electrical conductivity (dS m^{-1})	0.31 ± 0.03	0.92 ± 0.02
pH	7.5 ± 0.10	7.2 ± 0.21
Calcium carbonate (%)	13.4 ± 1.1	5.3 ± 0.51
Cation exchange capacity (mmol kg^{-1})	61.0 ± 3.55	50.8 ± 3.95
Total organic C (%)	0.47 ± 0.031	0.33 ± 0.07
Total N (mg g^{-1} soil)	0.39 ± 0.06	0.26 ± 0.04
Olsen P (μg g^{-1} soil)	3.9 ± 0.35	3.5 ± 0.16
NH_4OAc extractable K (μg g^{-1} soil)	105.0 ± 6.43	76 ± 3.06
$CaCl_2$ extractable SO_4^{2-} (μg g^{-1} soil)	9.4 ± 0.67	7.7 ± 0.35
AB-DTPA extractable Zn (μg g^{-1} soil)	2.2 ± 0.17	2.7 ± 0.10
AB-DTPA extractable Fe (μg g^{-1} soil)	2.8 ± 0.15	1.4 ± 0.06
AB-DTPA extractable Cu (μg g^{-1} soil)	0.8 ± 0.16	0.7 ± 0.06
AB-DTPA extractable Mn (μg g^{-1} soil)	23.8 ± 0.26	26.2 ± 0.55

C, carbon; *N*, nitrogen; *P*, phosphorus; *K*, potassium; SO_4^{2-}, sulfate; *Zn*, zinc; *Fe*, iron; *Cu*, copper; *Mn*, manganese. Data are mean ± standard error, $n = 3$

3.2 Soil Microbial Parameters

The rate of CO_2-C and cumulative CO_2-C (ΣCO_2-C) evolution differed significantly with the soil type, being higher in Missa than the Kahuta soil (Table 3), while the ratio of $\sum CO_2$-C to microbial biomass carbon was significantly lower in the Missa soil. Addition of organic amendments resulted in a substantial increase in CO_2-C evolution in the soils in comparison with the control. The highest rate of CO_2-C evolution and the ΣCO_2-C was found in PL-treated soils and the lowest in FYM-treated soils (Fig. 1). The ratio of ΣCO_2-C to microbial biomass C was significantly lower in FYM in comparison with SF- and PL-amended soils. The microbial biomass C and microbial biomass S contents also differed significantly between the soils and were higher in Missa than the Kahuta soil irrespective of the organic amendments' addition (Table 4). The organic amendments achieved a significant prompting impact on the above-mentioned microbial parameters. However, the increase in soil MBC was highest in PL-amended soils, while the MBS was highest in the SF-amended soils (Figs. 2 and 3). During the incubation period of 56 days, the maximum increase in the MBC and MBS contents was observed on the 14th day of incubation which later on decreased gradually until the end of incubation. The box and whisker plots visualize the distribution of the treatment effect on MBC and MBS in both soils in comparison with the control. The visualization is sorted on an average basis and the mean MBC and MBS values for each treatment are demonstrated by the middle line in each box. The box stretches from the first quartile to the third quartile, and the whiskers stretch at 1.5 inter-quartile range. There are also a number of outlier values, the points that are placed outside the whiskers.

The activities of soil enzymes, i.e., arylsulfatase activity and dehydrogenase activity, varied widely among the soils being higher in Missa compared with that in the Kahuta soil (Table 4). Organic amendments significantly increased ASA and DHA in the soils in comparison with the control. DHA was highest in the PL-amended soils, whereas ASA was highest in the SF-amended soils, with the overall trend varied as $PL > FYM > SF$ for DHA and $SF > PL > FYM$ for ASA. Both the enzyme activities increased gradually from the start of incubation. DHA reached its peak at 28 DOI while the ASA at 42 DOI and later on decreased until the end of incubation.

The dissolved organic carbon also varied significantly with the soils, higher in Missa than the Kahuta soil (Table 4). Among the organic amendments, PL caused more increase in DOC than the FYM and SF amendments in the following order: $PL > SF > FYM$. Opposite to the MBC and MBS, the DOC was significantly highest at 0 DOI in both the control and the treated soil, declined sharply until 14 DOI, and then increased gradually at 28 and 42 DOIs to get stable at 56 DOI. The ratio of microbial biomass C to microbial biomass S (MBC/MBS) varied significantly in both the soils (Table 5).

Table 2 Composition of organic amendments

Properties	Farmyard manure (FYM)	Poultry litter (PL)	Sugarcane filter cake (SF)
Moisture content (%)	7.9 ± 0.23	41.2 ± 0.60	12.2 ± 0.46
Total organic C (%)	22.4 ± 0.75	31.8 ± 0.77	28.6 ± 1.61
Total N (mg g^{-1})	1.3 ± 0.76	2.9 ± 0.47	2.1 ± 0.35
Total P (mg g^{-1})	3.6 ± 0.10	12.7 ± 0.29	9.7 ± 0.22
Total S (%)	0.13 ± 0.03	0.23 ± 0.02	0.31 ± 0.04
Total Zn (µg g^{-1})	2.5 ± 0.10	18.5 ± 0.26	0.7 ± 0.03
Total Cu (µg g^{-1})	0.4 ± 0.02	0.7 ± 0.01	0.7 ± 0.06
Total Fe (µg g^{-1})	63.4 ± 1.48	5.6 ± 0.04	5.0 ± 0.1
Total Mn (µg g^{-1})	7.6 ± 0.18	1.5 ± 0.06	1.1 ± 0.11
Total Cd (µg g^{-1})	1.3 ± 0.02	1.7 ± 0.03	1.5 ± 0.05
Total Cr (µg g^{-1})	1.5 ± 0.03	3.2 ± 0.21	1.3 ± 0.04
Total As (µg g^{-1})	0.2 ± 0.17	0.4 ± 0.03	Nd
Total Ni (µg g^{-1})	3.7 ± 0.02	4.2 ± 0.23	Nd
Total Pb (µg g^{-1})	2.1 ± 0.07	2.9 ± 0.05	Nd
Total Hg (µg g^{-1})	0.04 ± 0.002	0.02 ± 0.001	Nd
Total organic C/total N	13.9 ± 0.46	11.1 ± 0.50	14.0 ± 0.36
Total organic C/total P	4.8 ± 0.09	2.5 ± 0.06	2.9 ± 0.08
Total organic C/total S	133.07 ± 0.63	138.26 ± 0.86	92.26 ± 1.52

Nd, not detectable; *C*, carbon; *N*, nitrogen; *P*, phosphorus; *S*, sulfur; *Zn*, zinc; *Cu*, copper; *Fe*, iron; *Mn*, manganese; *Cd*, cadmium; *Cr*, chromium; *As*, arsenic; *Ni*, nickel; *Pb*, lead; *Hg*, mercury. Data are mean ± standard error, $n = 3$

MBC/MBS was higher in the Missa soil as compared with that in the Kahuta soil. Among the organic amendments, the ratios were higher in FYM-treated soils followed by SF (except DHA/MBC) and lowest in the PL-treated soils. The ratios of MBC to MBS, dehydrogenase activity to microbial biomass C (DHA/MBC), and arylsulfatase activity to microbial biomass C (ASA/MBC) also varied significantly in both the soils (Table 5). MBC/MBS was high in the Missa soil whereas the Kahuta soil had high DHA/MBC and ASA/MBC values. Among the organic amendments, the ratios were higher in FYM-treated soils followed by SF (except DHA/MBC) and lowest in the PL-treated soils.

Table 3 Main effect of soils, organic amendments, sampling days, and their interaction on the rate of CO_2-C evolution, cumulative CO_2-C (ΣCO_2-C), and cumulative CO_2-C to microbial biomass carbon (ΣCO_2-C/MBC) ratio

Main Effects	Rate of CO_2-C evolution (µg g^{-1} soil day^{-1})	ΣCO_2-C (µg g^{-1} soil)	ΣCO_2-C/MBC
Soils (S)			
Missa	$48.3 \pm 14.9a$	$2534.4 \pm 871.7a$	$10.4 \pm 1.2b$
Kahuta	$40.6 \pm 12.6b$	$2094.8 \pm 697.1b$	$10.8 \pm 1.4a$
LSD	0.54	29.4	0.30
Organic amendments (O)			
Control	$25.9 \pm 2.8d$	$1270.7 \pm 90.4d$	$09.7 \pm 0.6b$
FYM	$38.4 \pm 3.3c$	$1950.5 \pm 228.9c$	$09.1 \pm 0.5c$
PL	$58.7 \pm 5.6a$	$3155.0 \pm 320.8a$	$11.8 \pm 0.4a$
SF	$54.8 \pm 5.5b$	$2882.2 \pm 329.0b$	$11.6 \pm 0.5a$
LSD	0.77	41.52	0.42
Analysis of variance (*p* value)			
S	0.0000	0.0000	0.0218
O	0.0000	0.0000	0.0000
S × O	0.0000	0.0000	0.0319

FYM, farmyard manure; *PL*, poultry litter; *SF*, sugarcane filter cake. Letters indicate significant differences at $p < 0.05$ within the column (LSD test). Data are mean ± standard error, $n = 3$
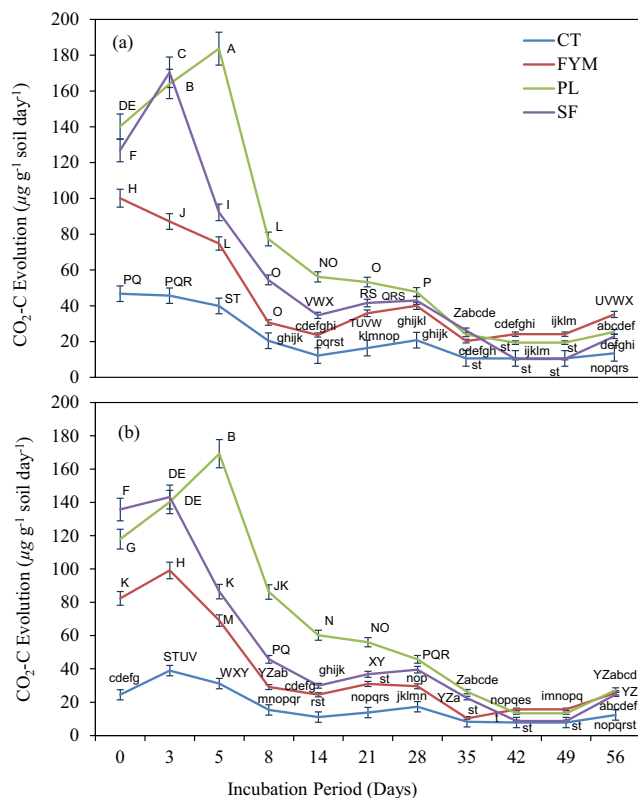
Fig. 1 Effect of organic amendments (CT, control; FYM, farmyard manure; PL, poultry litter; SF, sugarcane filter cake) on CO_2-C evolution per day in Missa and Kahuta soils, respectively. Different letters show a statistically significant difference ($p \leq 0.05$) among the treatments. Error bars represent 95% confidence intervals for $n = 3$

3.3 Plant-Available Sulfate and Sulfur Fractions

Available sulfur represented by 0.01 M $CaCl_2$ extractable sulfate was considerably higher in the Missa soil (14.2 μg g^{-1} soil) in comparison with that in the Kahuta soil (11.4 μg g^{-1} soil) (Fig. 4). Organic amendments caused a significant increase in the available sulfur contents in both the soils in the following order: SF > PL > FYM. The available S increased gradually in the amended soils from 0 DOI until the end of incubation (56 DOI). Both the soils also varied in terms of total S content and different S fractions (Table 6). Missa soil showed a significant increase in sulfur fractions and total S content as compared with the Kahuta soil. The net increase in inorganic sulfur fractions was observed in almost all the organic amendments. The highest increase was observed in SF-treated soil followed by PL- and FYM-treated soils. In addition to inorganic S, the organic S fractions (C-bonded S and ester sulfate S) also increased at the start of incubation immediately after the organic amendment addition. Water-soluble S and adsorbed S contents gradually increased with the incubation period and were highest at 56 DOI. Acid-soluble sulfate, carbon-bonded S, and ester sulfate S fractions were highest initially and decreased with the progress of incubation (0 DOI to 56 DOI). Also, the adsorbed S fraction increased gradually

with the incubation period and found highest at the end of the incubation experiment (56 DOI). A strong positive correlation of plant-available sulfate was observed with the water-soluble S ($R^2 = 0.94$), C-bonded S ($R^2 = 0.89$), and total S ($R^2 = 0.88$) contents in the soils (Fig. 5). Also, the positive correlation of plant-available sulfate was found with the adsorbed S fraction ($R^2 = 0.72$) in the soils. A fair positive relationship of plant-available sulfate with the acid-soluble S ($R^2 = 0.57$) and ester sulfate S ($R^2 = 0.47$) fractions also existed in the soils.

4 Discussion

4.1 CO_2-C Evolution and Enzyme Activities

Although the organic amendments, viz., farmyard manure, poultry litter, and sugarcane filter cake, significantly increased CO_2-C and ΣCO_2-C evolution from the soils, the effect of PL was more prominent than the SF and FYM amendments (Bhoi and Mishra 2012). The higher contents of total organic C, dissolved organic C, and other essential nutrients in PL might have stimulated the growth and activity of microbial populations present in the soils (Malik et al. 2013) and thus resulted in increased soil respiration (Premanandarajah and Shanika 2015). The rate of CO_2-C evolution increased in the soils immediately after the addition of organic amendments. This shows the availability of organic C as an energy source to the soil microorganism soon after the addition of organic amendments (Duong et al. 2009). Since the soils were deficient in organic matter and other essential nutrient elements, the addition of organic substrates enhanced microbial activity to initiate decomposition process, which is the prime function of soil microorganisms. The rate of CO_2-C evolution was highest at the early stage of the incubation showing more availability of organic C to the starved microbial population. At the later stage of incubation, the CO_2-C evolution gradually decreased, with the lesser availability of labile organic compounds due to mineralization, and became at par with the untreated control (Gilani and Bahmanyar 2008).

Soil enzyme activities, viz., dehydrogenase and arylsulfatase also increased by the addition of organic amendments. Organic compounds in the soils are oxidized through the dehydrogenase enzyme; therefore, the activity of this enzyme is intensified by the addition of organic amendments. The added organic materials may contain intra- and extracellular enzymes and also stimulate soil microorganisms to release enzymes (Navnage et al. 2018; Scherer et al. 2011). Therefore, it is likely that PL amendment resulted in higher stimulation of DHA due to its high TOC and DOC contents as previously reported by Scherer et al. (2011). Arylsulfatase enzyme is responsible for sulfur mineralization in soil due to its involvement in the hydrolysis of organic S compounds (Chen et al. 2019). A significant increase in ASA at the early

Table 4 Main effect of soils, organic amendments, sampling days, and their interaction on dissolved organic carbon (DOC), microbial biomass carbon (MBC), microbial biomass sulfur (MBS), dehydrogenase activity (DHA), and arylsulfatase activity (ASA) in soils

Main effects	DOC ($\mu g\ g^{-1}$ soil)	MBC ($\mu g\ g^{-1}$ soil)	MBS ($\mu g\ g^{-1}$ soil)	DHA (μg INF g^{-1} soil)	ASA (μg p-nitrophenol $g^{-1}\ h^{-1}$)
Soils (S)					
Missa	$112.0 \pm 29a$	$214.4 \pm 65.9a$	$3.89 \pm 1.3a$	$9.89 \pm 4.7a$	$8.74 \pm 4.5a$
Kahuta	$78.4 \pm 23b$	$150.7 \pm 52.4b$	$2.93 \pm 1.1b$	$7.48 \pm 3.4b$	$6.85 \pm 3.6b$
LSD	2.72	3.69	0.03	0.16	0.12
Organic amendments (O)					
Control	$65.3 \pm 14.9d$	$119.8 \pm 27.4d$	$1.88 \pm 0.5d$	$4.59 \pm 0.6d$	$3.62 \pm 0.5d$
FYM	$83.7 \pm 17.1c$	$166 \pm 31.2c$	$2.98 \pm 0.9c$	$9.82 \pm 3.6b$	$8.08 \pm 3.3c$
PL	$126.4 \pm 29.5a$	$234.9 \pm 69.7a$	$4.090.6 \pm b$	$11.30 \pm 4.7a$	$10.30 \pm 4.1a$
SF	$105.3 \pm 20.2b$	$209.5 \pm 63.9b$	$4.70 \pm 0.9a$	$9.01 \pm 3.7c$	$9.19 \pm 4.2b$
LSD	3.84	5.22	0.04	0.22	0.16
Sampling days (D)					
0 day	$106.6 \pm 34.1a$	$161.7 \pm 45.4b$	$2.79 \pm 1.0e$	$4.86 \pm 0.8e$	$3.68 \pm 0.6e$
14 days	$81.6 \pm 30.8c$	$241.9 \pm 89a$	$4.24 \pm 1.4a$	$7.47 \pm 2.5d$	$5.47 \pm 1.8d$
28 days	$96.5 \pm 29.1b$	$186.1 \pm 63.3b$	$3.67 \pm 1.3b$	$13.05 \pm 5.2a$	$10.20 \pm 4.1b$
42 days	$96.7 \pm 30.5b$	$167.7 \pm 49.5c$	$3.29 \pm 1.2c$	$10.08 \pm 3.6b$	$10.71 \pm 4.4a$
56 days	$81.6 \pm 27.9c$	$155.5 \pm 43.5d$	$3.07 \pm 1.1d$	$8.03 \pm 2.6c$	$8.96 \pm 3.5c$
LSD	4.29	5.83	0.05	0.25	0.19
Analysis of variance (p value)					
S	0.0000	0.0000	0.0000	0.0000	0.0000
O	0.0000	0.0000	0.0000	0.0000	0.0000
D	0.0000	0.0000	0.0000	0.0000	0.0000
S × O	0.0000	0.0000	0.0000	0.0000	0.0000
O × D	0.0121	0.0000	0.0000	0.0000	0.0000
S × O × D	0.9726	0.0215	0.0000	0.0000	0.0000

FYM, farmyard manure; *PL*, poultry litter; *SF*, sugarcane filter cake. Letters indicate significant differences at $p < 0.05$ within the column (LSD test). Data are mean $\pm$ standard error, $n = 3$

stages of incubation (i.e., within the first 4 weeks) was observed in soils amended with SF, followed by PL and FYM amendments. This could be associated with more availability of oxidizable S from the SF amendment due to its high organic S contents (Saviozzi et al. 2002). The trend of available SO_4^{2-} and ASA observed in our incubation study indicates that low SO_4^{2-} concentration in the soil in the initial phase of incubation stimulated higher ASA, whereas the high SO_4^{2-} concentration at the later stage of incubation induced a sharp decrease in ASA. Thus, arylsulfatase activity might be under the feedback control mechanism in line with inorganic SO_4^{2-} concentration in soil as reported by Siwik-Ziomek et al. (2016).

4.2 Microbial Biomass C and Microbial Biomass S

In the current investigation, a greater increase in soil microbial biomass C was induced by PL in comparison with SF and FYM amendments because of its high total organic C, total N, and P contents. Similarly, the lower C to N and C to P ratios of the PL were also probably the reason for the rapid

degradation of PL. Consequently, the MBC increased more rapidly in PL treatment as stated by Premanandarajah and Shanika (2015). The preliminary increase in MBC in the amended soils reflects the decomposition of easily degradable substrates contained in the organic amendments during the early phase of incubation. Later on, the available labile C and energy supply decreased due to the decomposition of the amendments and thus resulted in a significant decrease in MBC (Chowdhury et al. 2000).

Microbial biomass sulfur is one of the chief factors leading to S transformations in soil. The study shows that magnitude of MBS is affected by the addition of organic inputs in the soils. A marked increase in MBS induced by organic manure addition to the soil has been reported in some previous studies. Wu et al. (1993) revealed that 20 to 33% of the total sulfur contents in barley and oilseed rape residues were converted into MBS within 15 days of their incorporation into the soil. Yang et al. (2007) reported a massive rise in MBS at high doses of FYM application. Howlader et al. (2008) indicated that the rice straw amendment contributed to the highest

Fig. 2 Effect of organic amendments (CT, control; FYM, farmyard manure; PL, poultry litter; SF, sugarcane filter cake) on microbial biomass carbon and microbial biomass sulfur in Missa (a, c) and Kahuta (b, d) soils, respectively. Different letters show a statistically significant difference ($p \leq 0.05$) among the treatments. Error bars represent 95% confidence intervals for $n = 3$
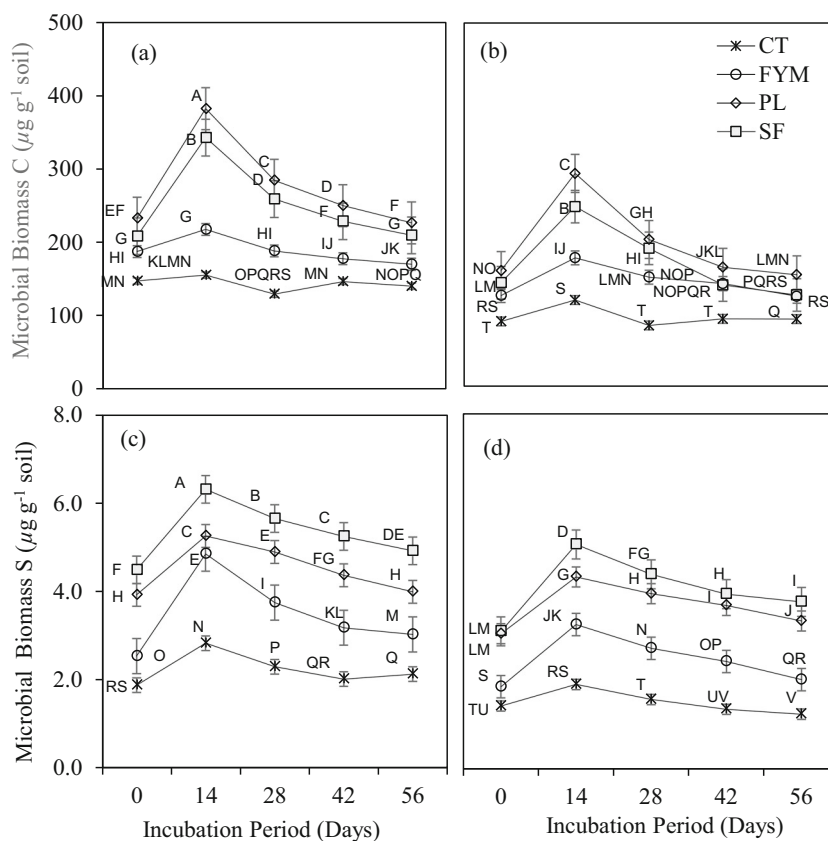
amount of MBS in soils as compared with PL, dustbin waste, and sewage sludge application. The above studies indicated that sulfur contents in organic amendments greatly influence soil MBS. Likewise, an increase in MBS in the present study is in line with the total S contents of the organic amendments.

4.3 S Fractions and their Distribution

Sulfur fractions and their relationship to available S contents in soil in response to organic amendments have been less explored particularly in alkaline calcareous soils. Changes in sulfur fractions in organically amended soils started at the early phase and continued until the end of incubation. This indicates the non-static behavior of organic S fractions in the soils (Kertesz et al. 2007). Water-soluble and adsorbed S fractions, which are recognized as the main components of plant-available S, remained lowest at the start of incubation but increased gradually to become higher at the end of incubation. This increase in available S might be partly due to acidulation effect of the added organic amendments on insoluble S fractions in the soils to release available SO_4^{2-}. Khan and Joergensen (2009) and Khan et al. (2019) reported the similar acidulation effect of composted organic material on P solubility. The increase in available S at the later stage of incubation might also be because of the mineralization of added organic sulfur into inorganic sulfate.

The available SO_4^{2-} contents were higher in SF-treated soils as compared with other organic amendments that could be associated with the sulfur contents of the organic amendments. According to Reddy et al. (2001), sulfur mineralization capacity of soil depends on the sulfur content of the added decomposable material. The C to S ratio of SF amendment was lowest among all the amendments and thus, it could be the possible reason for higher sulfate mineralization in SF-amended soils as compared that in PL and FYM. Our results support the findings of Lucheta and Lambais (2012) who reported that sulfate is either released or tied up in organic form depending upon C to S ratio of the added material. The C to S ratio of less than 200 resulted in the release of sulfate whereas more than 200 immobilized the sulfate in soils.

The concentration of adsorbed S extracted with KH_2PO_4 was lower in the early phase of incubation but later on, it increased with the progress of incubation. These observations are consistent with the statement of Haque and Walnsley (1973) who presented a positive correlation between the sorption of SO_4^{2-} and organic compounds in soil. On the other hand, Singh and Johnson (1986) observed a negative correlation, and Balik et al. (2009) reported a decrease in sulfate sorption due to increased sorption of organic anions on to soil colloids after organic manure addition. Sulfate adsorption is pH-dependent phenomena in soil and increases with a decreasing soil pH due to protonation of soil colloid
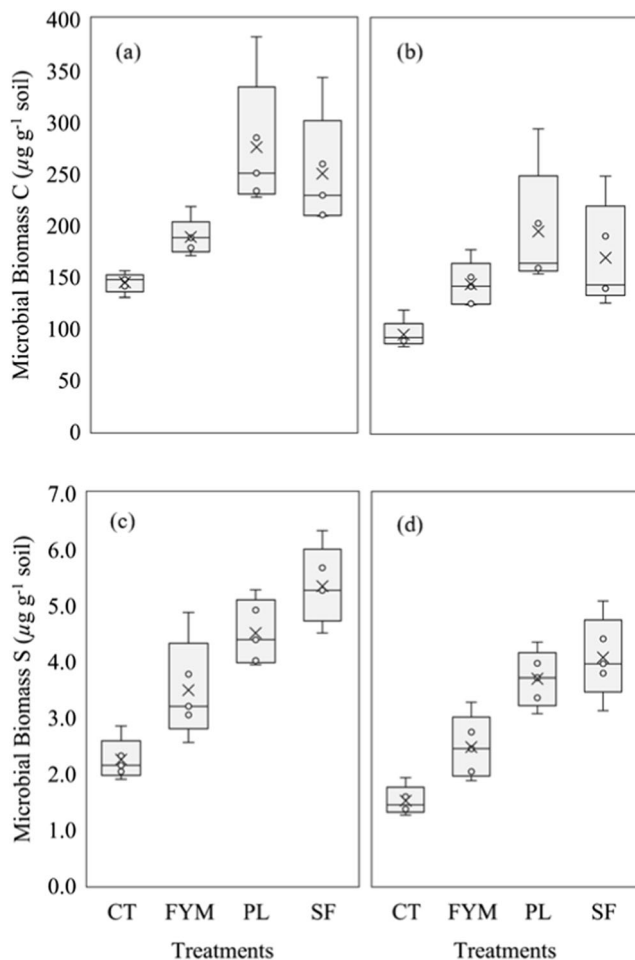
Fig. 3 The box and whisker plots visualizing the distribution of the treatment's effect (CT, control; FYM, farmyard manure; PL, poultry litter; SF, sugarcane filter cake) on microbial biomass carbon and microbial biomass sulfur in Missa (a, c) and Kahuta (b, d) soils. The visualization is sorted on an average basis and the mean MBC and MBS values for each treatment are demonstrated by the middle line in each box. The box stretches from the first quartile to the third quartile, and the whiskers stretch 1.5 inter-quartile range. There are also several outlier values, the points that are placed outside the whiskers

Table 5 Main effect of soils, organic amendments, sampling days, and their interaction on a ratio of microbial biomass carbon/microbial biomass S (MBC/MBS), dehydrogenase activity/microbial biomass carbon (DHA/MBC), and arylsulfatase activity/microbial biomass carbon (ASA/MBC)

Main effects	MBC/MBS	DHA/MBC	ASA/MBC
Soils (S)			
Missa	$96.3 \pm 26.8a$	$0.046 \pm 0.02b$	$0.041 \pm 0.01b$
Kahuta	$35.1 \pm 9.8b$	$0.050 \pm 0.02a$	$0.046 \pm 0.02a$
LSD	2.02	0.0015	0.0012
Organic amendments (O)			
Control	$44.2 \pm 24.6d$	$0.039 \pm 0.007d$	$0.030 \pm 0.01d$
FYM	$82.6 \pm 45.3a$	$0.060 \pm 0.02a$	$0.050 \pm 0.02a$
PL	$62.3 \pm 27.3c$	$0.048 \pm 0.02b$	$0.045 \pm 0.02c$
SF	$73.7 \pm 35.8b$	$0.045 \pm 0.017c$	$0.047 \pm 0.03b$
LSD	2.85	0.002	0.001
Sampling days (D)			
0 day	$77.9 \pm 43.7a$	$0.029 \pm 0.01e$	$0.022 \pm 0.01d$
14 days	$63.8 \pm 30.9b$	$0.032 \pm 0.03d$	$0.023 \pm 0.004d$
28 days	$60.0 \pm 33.0c$	$0.069 \pm 0.02a$	$0.053 \pm 0.01c$
42 days	$63.3 \pm 36.5b$	$0.059 \pm 0.01b$	$0.062 \pm 0.02a$
56 days	$63.5 \pm 38.5b$	$0.052 \pm 0.01c$	$0.056 \pm 0.01b$
LSD	3.19	0.002	0.0019
Analysis of variance (p value)			
S	0.0000	0.0000	0.0000
O	0.0000	0.0000	0.0000
D	0.0000	0.0000	0.0000
S × O	0.0000	0.0000	0.0000
S × D	0.0000	0.0000	0.0000
O × D	0.0000	0.0186	0.0000
S × O × D	0.0023	0.7708	0.0000

FYM, farmyard manure; *PL*, poultry litter; *SF*, sugarcane filter cake. Letters indicate significant differences at $p < 0.05$ within the column (LSD test). Data are mean ± standard error, $n = 3$

surface functional groups. Hence, low content of adsorbed SO_4^{2-} observed at the start of incubation and its increase at a later stage of incubation as observed in our study might be linked to the development of SO_4^{2-} sorption sites in the soils (Förster et al. 2012). However, the present study is not conclusive in this regard and further investigations are required to verify this observation.

Compared with water-soluble and adsorbed SO_4^{2-}, acid-soluble sulfate extractable with 1 M HCl was initially higher in the soils and further increased with organic amendments addition. A larger concentration of HCl extractable sulfur indicates the presence of water-insoluble but acid-soluble sulfates. This sulfur fraction is likely to be present in co-precipitated and co-crystallized forms with $CaCO_3$ in calcareous soils (Hu et al. 2005). Organic amendments significantly

increased HCl-soluble S fraction in both the soils at the earlier stages of incubation but decreased later on. In Canadian and Australian soils, acid-soluble SO_4^{2-} (occluded S) accounted for up to 42 to 93% of the total soil S (Hu et al. 2005), whereas in the current study, this fraction accounted to be 23.6% and 20.7% in Missa and Kahuta soils, respectively. These findings suggest that acid-soluble S exists as a significant sulfur fraction in calcareous soils of semi-arid/arid subtropical regions but is generally neglected in plant nutrient management.

Organic S was the dominant sulfur fraction in the soils used in the present study as has also been reported earlier (Basumatary et al. 2018; Yang et al. 2007). Incorporation of organic material strongly enhanced total sulfur and organic sulfur fractions in the soils (Luo et al. 2014), depending upon their type and composition (Reddy et al. 2001). The highest rise in organic S was monitored in SF-amended soils at the
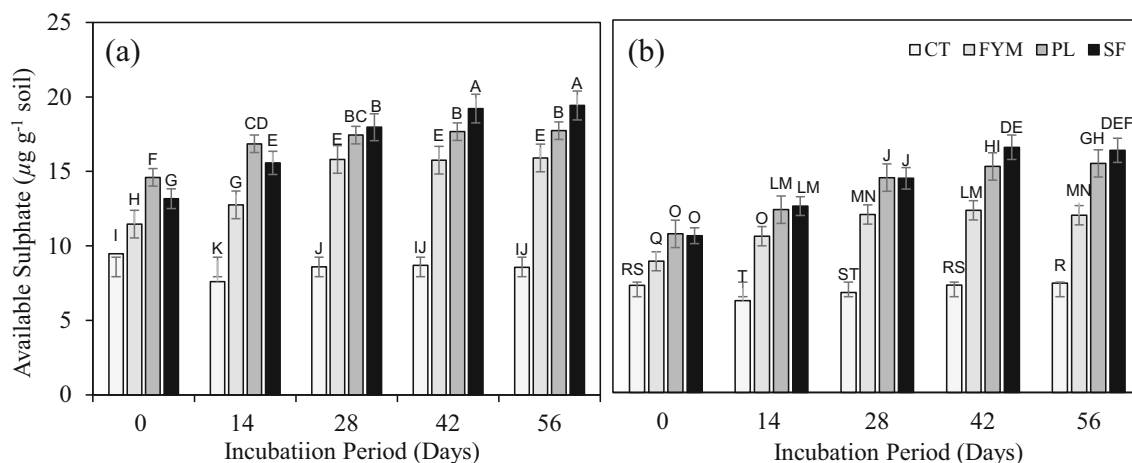
Fig. 4 Effect of organic amendments (CT, control; FYM, farmyard manure; PL, poultry litter; SF, sugarcane filter cake) on available sulfate in Missa and Kahuta soils, respectively. Different letters on various treatment bars show a statistically significant difference ($p \leq 0.05$) among them. Error bars represent 95% confidence intervals for $n = 3$

start of incubation which later on decreased with the progress of time due to the mineralization (Saren et al. 2016). Among the organic S fractions, carbon-bonded S was the dominant fraction because, in systems where SO_4^{2-} availability is low, the C-bonded S dominates and is linked with the soil carbon contents. Inputs of organic amendment rich in carbon facilitate

Table 6 Main effect of soils, organic amendments, sampling days and their interaction on sulfur (S) fractions in soils

Main effects	Water-soluble S (μg g^{-1} soil)	Adsorbed S (μg g^{-1} soil)	Acid-soluble S (μg g^{-1} soil)	Carbon-bonded S (μg g^{-1} soil)	Ester-bonded S (μg g^{-1} soil)	Organic S (μg g^{-1} soil)	Total S (μg g^{-1} soil)
Soils (S)							
Missa	11.8 ± 3.6a	3.62 ± 0.9a	25.4 ± 3.2a	56.5 ± 5.6a	22.8 ± 3.1a	79.3 ± 7.9a	107.6 ± 10a
Kahuta	9.5 ± 3b	2.10 ± 0.5b	19.6 ± 3.0b	49.8 ± 6.1b	15.4 ± 2.0b	65.2 ± 7.5b	94.4 ± 9.5b
LSD	0.16	0.08	0.42	0.78	0.38	0.69	1.62
Organic amendments (O)							
Control	6.7 ± 0.9d	1.98 ± 0.6d	19.9 ± 2.9d	46.1 ± 4.3d	16.9 ± 4.1d	62.9 ± 8.3d	87.8 ± 7.2c
FYM	9.6 ± 1.5c	2.53 ± 0.7c	20.9 ± 3.2c	52.3 ± 4.6c	18.6 ± 4.4c	70.9 ± 8.5c	97.3 ± 7.1b
PL	12.7 ± 2.5b	3.11 ± 0.9b	22.2 ± 3.5b	55.2 ± 4.7b	21.2 ± 4.7a	76.4 ± 9.0b	108.5 ± 8.6a
SF	13.6 ± 3.2a	3.83 ± 1.1a	27.1 ± 3.6a	59.1 ± 5.7a	19.6 ± 4.2 b	78.7 ± 8.9a	110.4 ± 6.5a
LSD	0.22	0.11	0.59	1.11	0.54	0.98	2.29
Sampling days (D)							
0 day	8.6c (1.7)	2.61 ± 0.9c	24.1 ± 4.4a	56.6 ± 7.1a	21.2 ± 5.4a	77.8 ± 11.2a	101.6 ± 12.9a
28 days	11.3b (3.7)	2.82 ± 1.0	22.2 ± 4.0b	53.1 ± 5.5b	18.6 ± 4.4b	71.7 ± 8.8b	100.8 ± 10.8a
56 days	12.0a (3.8)	3.15 ± 1.2a	21.3 ± 4.0c	49.8 ± 5.9c	17.5 ± 2.9c	67.3 ± 8.8c	100.6 ± 11.7a
LSD	0.19	0.10	0.50	0.96	0.47	0.85	1.98
Analysis of variance (*p* value)							
S	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
O	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
D	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.6038
S × O	0.0000	0.2503	0.2491	0.8403	0.9975	0.8044	0.8930
S × D	0.0883	0.5721	0.5752	0.0023	0.0000	0.0007	0.6938
O × D	0.0000	0.3086	0.3111	0.0046	0.0001	0.0027	0.6386
S × O × D	0.9110	0.9999	0.9999	0.9200	0.4297	0.9995	0.6760

FYM, farmyard manure; *PL*, poultry litter; *SF*, sugarcane filter cake. Letters indicate significant differences at *p* < 0.05 within the column (LSD test). Data are mean ± standard error, *n* = 3

Fig. 5 Relationship between available sulfate (SO$_4^{2-}$) and (a) water-soluble S, (b) adsorbed S, (c) acid-soluble S, (d) C-bonded S, (e) ester sulfate S, and (f) total sulfur

(a) y = 1.1279x + 0.7957
R² = 0.9452

(b) y = 2.8219x + 4.7294
R² = 0.726

(c) y = 0.6187x - 1.0098
R² = 0.573

(d) y = 0.5544x - 16.694
R² = 0.8867

(e) y = 0.585x + 1.6363
R² = 0.4799

(f) y = 0.283x - 15.787
R² = 0.8833
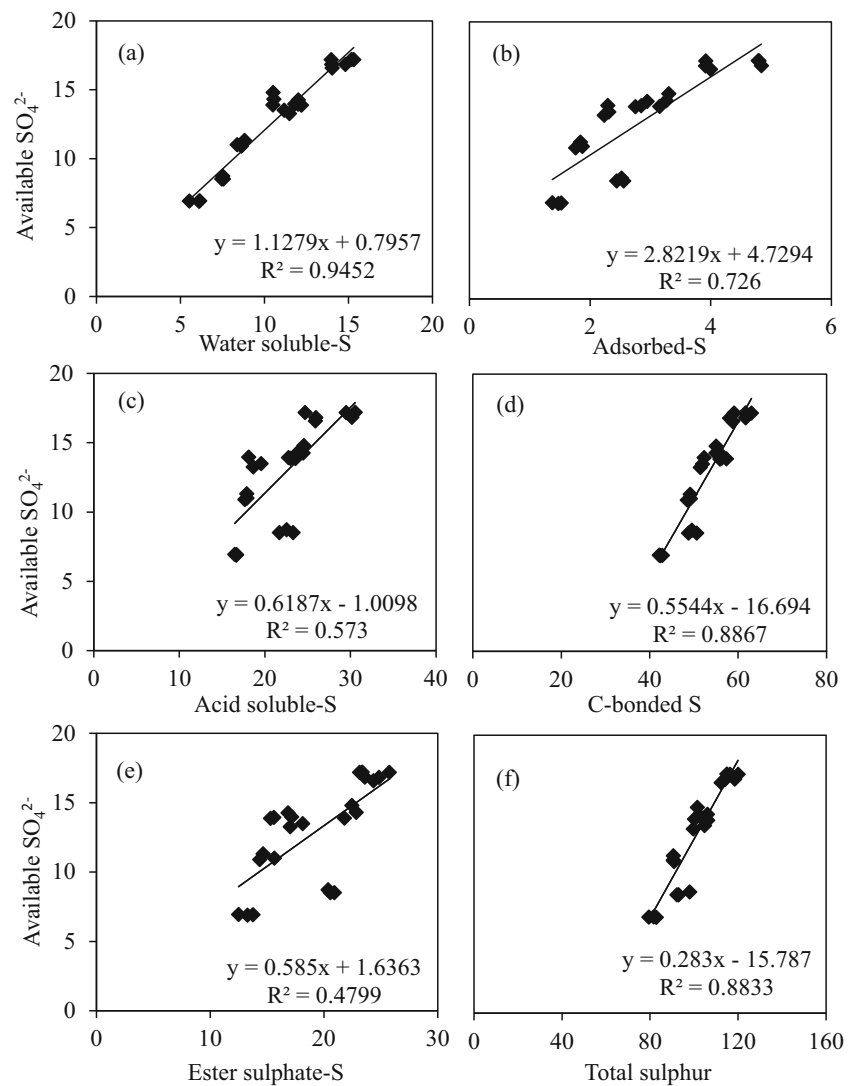
the accumulation of C-bonded S in soils (Knights et al. 2000). In our study, the application of organic amendments significantly increased C-bonded S fraction in both the soils in the following order SF > PL > FYM in the early phase of incubation. Zhao et al. (2006) revealed that in animal waste, carbon-bonded S counted for 80% of the total S, whereas in plant residues, it accounted for 60–90% of the total S. Solomon et al. (2011) observed that incorporation of sugarcane residues resulted in an initial increase of 40% in organo-sulfur compounds directly bonded to C (C-bonded S) just within 3 days of incubation. However, later on, this fraction decreased to 13% after 365 days of incubation. This decrease in C-bonded S fraction at a later stage of incubation might be due to biological oxidation of the C-S linkages to SO$_4^{2-}$ S or conversion into transient linkages as the ester sulfate. Zhao et al. (2006) and Solomon et al. (2011) suggested C-bonded S as the major source of sulfur mineralization in short-term incubation studies. The results of our incubation study regarding changes in organic S functions in soil due to organic

amendments addition are similar to the above-mentioned findings. Yang et al. (2007) observed a significant increase in C-bonded S, organic S, and total S contents in the soil after FYM application. Xu et al. (2016) reported that long-term use of organic manures significantly facilitated the accumulation of C-bonded S, residual S, and increased total and organic S contents in soils.

On the other hand, increase in ester sulfate S concentration in the amended soils at the start of incubation agrees with the studies where an increase in oxidizable S fraction (ester S) in the initial phase of organic materials decomposition was observed in the soil (Blum et al. 2013; Ghani et al. 1993). Solomon et al. (2011) found that the addition of inorganic sulfate facilitates the accumulation of ester sulfate S in soil. Thus, it can be presumed that an increase in the ester sulfate S fraction observed at the start of incubation might be the response of increased available sulfate contents in the soils. With time and further cycling, a proportion of ester sulfates is disseminated into the C-bonded S and is strongly influenced

by fluxes in soil microbial biomass and its activity but lesser by the sulfate content (Förster et al. 2012).

5 Conclusion

The study provides evidence of the strong influence of organic amendments on microbial activity, sulfur fractions, and their re-distribution in soil. The effect of organic amendments on study parameters varied depending upon their quality and composition. Total organic carbon, labile organic carbon, total sulfur, and extractable sulfur were the main components in organic amendments which significantly influenced sulfur fractions and their availability in soil. Overall, the organic amendments increased available sulfate in the following order: sugarcane filter cake > poultry litter > farmyard manure, and facilitated the accumulation of C-bonded S in soils. Net increase in inorganic S fractions was observed with all the organic amendments but highest in sugarcane filter cake, followed by poultry litter and farmyard manure amended soils. Acid-soluble sulfate, C-bonded S, and ester-bonded S fractions were highest at the start of incubation and decreased with time due to mineralization by microbial activity and the acidulation effect contributing to water-soluble S fraction. Acid-soluble S (occluded S) exists as a significant S fraction in calcareous soils and may contribute to S availability in organic amended soils. The study further suggests that organic amendments might lead to the development of SO_4^{2-} sorption sites in the soil; however, investigations are required to verify this observation.

Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

Permission from Koh-e-Noor Sugar Mills The authors obtained permission from the responsible authority of the Koh-e-Noor Sugar Mills, Jauharabad, Khushab District, Punjab, Pakistan, for using their sugarcane filter cake in the study.

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