RESEARCH ARTICLE

The role of biochar and sulfur-modified biochar on soil water content, biochemical properties and millet crop under saline-sodic and calcareous soil

Musa al-Reza Taheri · Ali Reza Astaraei · Amir Lakzian · Hojat Emami

Abstract

Aims Soil microbial and enzymatic activity have important roles in soil health, nutrient cycle and plant growth. Biochar is an excellent biomass for increasing soil water content, but some biochar compounds, due to their high pH, are harmful to the soil. This study aimed to investigate the effect of biochar and sulfur-modified biochar on improving the soil water content and biochemical properties in a millet cropping system.

Methods The field experiment was performed in a completely random design with three replications. Experimental treatments included control, 15 t ha^{-1} sunflower biochar (B), and 15 t ha^{-1} sulfur-modified biochar (BS).

Results Biochar (B and BS) improved the soil water content by 47% and 35%, respectively, compared to the control. B and BS treatments improved soil biological properties, including microbial biomass carbon, basal respiration, and the activity of catalase, dehydrogenase, urease, and alkaline phosphatase enzymes compared to the control. The application of B and BS treatments decreased soil EC and sodium

adsorption ratios compared to the control. The highest (8.26) and lowest (7.83) values of soil pH were related to the B and BS treatments, respectively. Treatment of soil with B and BS enhanced soil dissolved organic carbon (90% and 33%), available nitrogen (74% and 48%), and available phosphorus (60% and 96%) compared to the control. The application of B and BS treatments enhanced plant nutrients (nitrogen, phosphorus, and potassium) and the leaf relative water content by 40% and 14%, respectively. Biochar (B and BS) improved millet yield by 58% and 115%, respectively, compared to the control.

Conclusion Sulfur increased the efficiency of biochar in amending saline and calcareous soil and enhanced millet yield by improving the chemical properties (pH and CEC) of biochar.

Keywords Enzyme activity · Microbial activity · Sunflower biochar · Sulfur · Soil water content · Salinity

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Introduction

The salinity of soil, which has reduced agricultural productivity, is one of the major agricultural issues facing the world today (Hassani et al. 2021). Soil salinity decreases plant yield by limiting the available water of the plant, increasing ion toxicity, reducing available elements in the soil, and decreasing the microbial and enzymatic activity of the soil (Safdar

et al. 2019). Soil microorganisms have a key role in the cycle of soil nutrients, so the reduction in microbial activities in saline soils can decrease the growth of plants (Andronov et al. 2012). Enhancing the microbial and enzymatic activity of the soil can help plants resist the negative effects of soil salinity, such as harmful ROS compounds (Sritongon et al. 2022).

Iran has an arid and semi-arid climate with an average annual precipitation of less than 250 mm. The lack of rainfall and high evaporation have caused Iran's agricultural lands to have problems such as saline-sodic and calcareous soil (Mousavi et al. 2022). More than 30% of agricultural land in Iran has a salinity problem (Emadi et al. 2018). More than 70% of Iran's agricultural lands have less than 1% organic matter (Rezaei et al. 2020). Lack of organic matter reduces the soil's water content and accelerates soil salinization (Wichern et al. 2020). To achieve sustainable agriculture, much attention should be given to soil organic matter. Sunflower is one of the main plants widely cultivated in Iran to produce cooking oil. Every year, a large amount of sunflower residue is left in the fields and buried in the soil by plows. A high C/N ratio of sunflower residues disrupts the cycle of soil nutrients and delays soil fertility (Babu et al. 2014). Additionally, sunflower residues have allelopathy compounds that can have a negative effect on the next crop (Flayyih and Almarie 2017). This source of organic carbon can be safely used to improve soil fertility with little management.

One of the materials that can help improve unfavorable soil conditions is biochar. Biochar is a black substance that is produced from organic residues during the pyrolysis process in the absence of oxygen (Wang et al. 2020). Many studies have been performed on the effect of biochar on soil quality. According to the findings of these studies, biochar increases (1) soil organic carbon (Glaser et al. 2002), (2) water holding capacity (Abel et al. 2013), (3) Soil aeration, (4) Nutrient availability, (5) Stimulation of soil microbial and enzymatic activity (Taheri et al. 2022), (6) Cation exchange capacity (CEC). Soil water retention is one of the advantages of biochar for soil. Biochar materials have a high water-holding capacity due to their high porosity, which depends on their raw materials (Streubel et al. 2011; Yu et al. 2013) reported that the application of 1–5% biochar in loamy soil increased the soil water holding capacity by 4–10%. The physical properties of biochar, such as

its water holding capacity (WHC), surface area, and surface functional groups, are closely related to its feedstock biomass (Ahmad et al. 2014).

The structure of the sunflower stem is fibrous. The micro and macro pores in the sunflower pith fiber have created much space that can hold a significant amount of water (Gluba et al. 2021). Water retention in plant residues is affected by physical and chemical processes; for example, the presence of micro and macro sized pores on the surface of the cell wall, which is occasionally open or closed, controls water retention (Almeida and Hernández 2006). In wood and composite technology, it is established that compounds such as cellulose and hemicellulose are hydrophilic and compounds such as lignin are hydrophobic, and the ratio of these two groups influences the characteristics of water retention (Pejic et al. 2008). Sunflower stalk pith has a large amount of pectin and cellulose and contains a small amount of lignin. The sunflower pith is different from the contents of other crops, such as sorghum and corn. Marechal and Rigal (1999) reported that sunflower stalk pith contains 45% cellulose and 3% lignin.

Biochar has many benefits for soil, but the high pH of biochar can be problematic for soil fertility, especially in terms of the availability of soil nutrients such as phosphorus (Cheng et al. 2018). Increasing the pH of soil increases microbial nitrification, which results in losses of nitrate and limited availability of ammonium, the preferred nitrogen source for plants (Xiao and Meng 2020). In some cases, the pH-increasing effect of biochar can create unfavorable conditions for plants, particularly in calcareous soils, which can result in yield losses (Xiao and Meng 2020; Bachmann et al. 2016). Some types of biochar have a high pH and cannot be used in soil; however, it appears that the pH of biochar can be reduced, and it can be used with substances such as sulfur. The application of biochar enriched with sulfur as a soil amendment results in improved soil CEC, organic matter, hydraulic conductivity, soil porosity, infiltration rate, and microbial activities (Hafeez et al. 2022). Biochar enriched with hydrogen sulfide gas increased the soybean plant's uptake of macro- and micronutrients as well as its biomass (Zhang et al. 2016). The modifications of biochar with inorganic acids show that this work has improved the physico-chemical properties of biochar, such as reducing pH and increasing its CEC, and that using acid-modified biochar in soil has improved the availability of soil nutrients (El-Sharkawy et al. 2022; Sahin et al. 2017). Millet is a C4 plant that is resistant to saline-sodic and

dry soil conditions and is considered a good fodder plant; this was the main reason for selecting millet as an experimental crop due to the agronomic livestock farming practices of the people in the study area.

This study hypothesizes that (1) sunflower stalk biochar improves the soil water content and biochemical properties. (2) Sulfur-modified biochar has a more significant effect on improving soil biological and chemical properties than biochar. This study aimed to investigate the effect of biochar and sulfur-modified biochar on soil water content and soil biological and chemical properties under saline-sodic and calcareous soils with millet plants.

Materials and methods

Biochar and sulfur-modified biochar preparation

The dried residue of sunflower stalks was used to produce biochar. The sunflower stalk was cut into pieces measuring 10 cm and produced biochar during the pyrolysis process at a temperature of 400 °C in the absence of oxygen (Fu et al. 2019). To produce sulfur-modified biochar (BS), sunflower biochar (B) was mixed with a 5% inorganic sulfur solution (purity of 80%). After 24 h, the sulfur and biochar suspensions were filtered and dried.

Biochar and sulfur-modified biochar were first air-dried and then dried in an oven at 70 °C for 24 h.

Analyses of biochar (B and BS)

Biochar (B and BS) was passed through a 0.5-mm sieve and used for chemical analysis. Carbon, hydrogen, nitrogen, and sulfur were measured by elemental analysis (trade name: CHNS, model: Vario EL III, manufacturer: Elementar, country: Germany). Alkaline cations (Ca^{2+}, Mg^{2+}, K^+, and Na^+), ash content, and CEC were measured according to the Singh et al. (2017) procedure (Table 1). The water-holding capacity of biochar was measured by the weighting method, as shown in Fig. 1. For Fourier transform infrared spectroscopy (FTIR) analysis, 10 mg of B and BS samples were mixed with 190 mg of spectroscopic-grade KBr and prepared for analysis after grinding. The FTIR measurements were performed with a Nicolet 6700 FTIR (Thermo Nicolet); the device was an AVATAR 370 FT-IR (manufacturer: Thermo Scientific, USA). The scans were obtained in the range of 400 to 4000 cm^{-1} with a resolution of 4 cm^{-1}.

Experimental site

This study was conducted in *Shahrabad* village (58° 29' N, 36° 0' E), located in the middle of the

Table 1 Characteristics of biochar (B) and sulfur-modified biochar (BS)

| Parameters	Unit	B	BS	Reference of measurement methods
ECe	(dS m^{-1})	6.74	6.81	Jackson (1973)
pH	-	10.31	7.56	Jackson (1973)
C	(%)	61	57	Walkley and Black (1934)
H	(%)	2.9	4.5	-
O	(%)	8.6	14	-
N	(%)	0.32	0.43	Subbaiah (1956)
S	(%)	0.11	1.4	-
Ash	(%)	22	17	-
Volatile matter	(%)	4.43	5.76	-
H:C	-	0.047	0.078	-
O:C	-	0.14	0.24	-
P	(g kg^{-1})	0.68	0.44	Olsen (1954)
K$^+$	(g kg^{-1})	22	10.8	Hanway and Heidel (1952)
Na$^+$	(g kg^{-1})	15.5	12.6	-
Ca^{2+}	(g kg^{-1})	54	42	-
Mg^{2+}	(g kg^{-1})	21.6	18	-
CEC	[Cmol (+) kg^{-1}]	36	39	-
Water holding capacity	(% w/w)	400	250	-
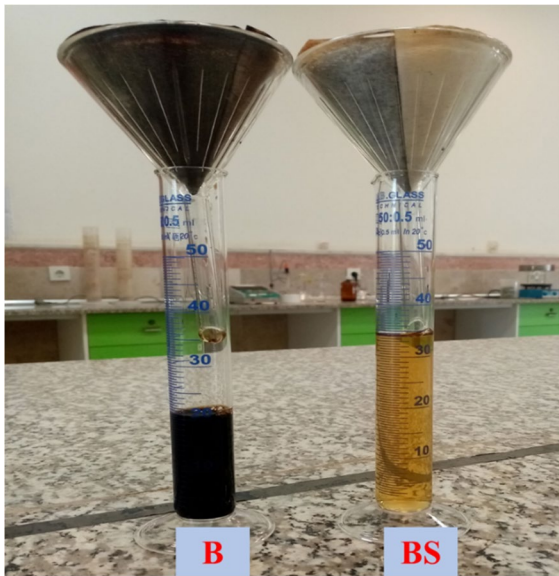
Fig. 1 Water holding capacity of biochar (B) and sulfur-modified biochar (BS)

Neyshabur plain of northeast Iran, in the summer of 2021 (June–August) under field conditions. The average rainfall in this area is less than 250 mm, and the summers are relatively hot (max 42 °C). The soil used in this experiment had a clay texture and had

problems with salinity, sodicity, and calcareousness. A soil sample was taken from 0 to 30 cm of surface soil depth for physicochemical analysis. Soil samples were taken on May 2, 2021 (Table 2).

Experimental design and treatments

This experiment was performed in a completely random design with three replications under field conditions. The experimental treatments were the control, 15 t ha^{-1} sunflower stem biochar (B), and 15 t ha^{-1} sulfur-modified biochar (BS). The experiment was carried out in a one-square-meter plot with a border distance of 50 cm. The B and BS treatments were added to the plot before planting (0–30 cm). In each plot, 10 millet (*Proso*) plants were grown as an experimental crop and cared for until the completion of postharvest analyses. The experimental plots were irrigated every 20 days with saline and sodic water (EC: 5 dS m^{-1} and SAR: 15). The millet plant was planted on 12 June 2021 and harvested on 4 November 2021 (Fig. 2).

Plant analysis

After the millet seeds ripened, the plant was harvested. The fresh weight of the plant was measured. The leaf relative water content (RWC) was calculated using the

Table 2 Physicochemical properties of experimental soil

Parameters	Unit	Values	Reference of measurement methods
Mechanical analysis	-	-	Bouyoucos (1962)
Sand	%	30	-
Silt	%	28	-
Clay	%	42	-
Texture	-	Clay	-
Chemical analysis	-	-	Richards et al. (1956)
pH	-	8.05	-
ECe	dS.m^{-1}	9.1	-
Ca^{2+}	meq.l^{-1}	12.6	-
Mg^{2+}	meq.l^{-1}	8.4	-
Na^{+}	meq.l^{-1}	81	-
SO4^{2-}	meq.l^{-1}	2.03	-
SAR	-	17.6	-
Calcium carbonate	%	18	-
Organic C	%	0.12	Walkley and Black (1934)
Available N	mg kg^{-1}	19.4	Subbaiah (1956)
Available P	mg kg^{-1}	6.2	Olsen (1954)
Available K	mg kg^{-1}	150	Hanway and Heidel (1952)
CEC	Cmol (+) kg^{-1} soil	27	Jackson (1973)

following formula. The fresh aboveground millet was harvested, dried in an oven at 70 °C for 48 h, and thereafter ground into powder for chemical analysis.

$$RWC = (FW - DW)/(TW - DW)$$

FW = leaf fresh weight, DW = leaf dry weight, TW = leaf turgid (saturated) weight

Soil analysis

Soil samples were taken from 0 to 30 cm in the experimental plots. The soil samples were divided into two parts: one part was kept for biological analysis at a temperature of 4 °C, and the other part of the soil sample was used for chemical analysis after drying and passing through a 2 mm sieve.

Soil water content (SWC)

After the germination and establishment of young plants, the experimental plots were irrigated, and samples were taken from the soil of the plots (0–20 cm) every day at 10:00 AM between the two irrigation times (irrigation period: 20 days). The soil was transferred to the laboratory and dried in an oven at 105 °C for 24 h to measure the soil water percentage. The soil water content was calculated by the difference between their fresh and dry weights (Novák et al. 2019).

$$SWC = (fresh\ weight - dry\ weight)/(dry\ weight)$$

Biological analysis of soil

The activity of alkaline phosphatase (ALP) was measured using the PNP-PO$_4$ method (p-nitro phenyl phosphate) and reported as µg PNP (p-nitro phenol)

in one gram of soil for one hour, which was measured by a spectrophotometer instrument (Tabatabai and Bremner 1969). Basal respiration (BR) was measured by the Anderson and Domsch (1990) method. Soil samples were kept in a desiccator at a temperature of 25 °C, and the amount of CO_2 produced was absorbed by sodium hydroxide and determined by titration with HCl. Microbial biomass carbon (MBC) was measured according to the fumigation and incubation method (Jenkinson 1976). Urease activity (UA) was measured by using a 10% urea solution as a substrate, and the amount of NH$_4$ released was determined by the colorimetric method (Hoffmann and Teicher 1961) at 578 nm by a spectrophotometer (Model: Halo XB-10, Company: Dynamic, Country: UK). Catalase activity (CAT) was determined by titration using Kmno$_4$ (Quan et al. 1986). The activity of dehydrogenase (DHA) was determined according to the triphenyl tetrazolium chloride (TTC) method and reported as µg triphenyl formazan (TPF) in one gram of soil for 24 h (Burns 1978).

Chemical analysis of soil

The dissolved organic carbon (DOC) was determined using the ferrous ammonium sulfate titration method (Herbert and Bertsch 1995). In this method, 0.5 M potassium sulfate was used for DOC extraction, a 0.45 µm filter was used to filter the extract, 0.0417 M potassium dichromate was used as an oxidizer, and 0.01 M ferrous ammonium sulfate was used for titration. The Olsen method was used to determine available phosphorus (AP), which involved extracting AP with 0.5 M sodium bicarbonate, coloring the extract blue with standard phosphorus colorimetric solutions (potassium antimony tartrate, ammonium heptamolybdate, and ascorbic

Fig. 2 Field experiment

acid), and measuring the blue color with a spectrophotometer at 660 nm (Olsen 1954). Available nitrogen (AN) was measured using the colorimetric method (Øien and Selmer-Olsen 1980). The available nitrogen was extracted by KCl solution, and after coloring with phenol and sodium hypochlorite, the green color was measured at 510 nm. Soil ECe and pH, soluble cations, and anions were measured in soil-saturated paste extract (Richards et al. 1956).

Statistical analysis

JMP software used one-way ANOVA and Tukey's HSD test with a 95% confidence level to analyze significant differences between treatments. The relative importance analysis was performed by the R package "relaimpo" to find the key soil properties that control millet yield.

Results

Characteristics of biochar (B) and sulfur-modified biochar (BS)

Modification of biochar with sulfur increased sulfate ions in the BS treatment and decreased its pH by approximately 3 units compared to the B treatment (Table 1). Sulfur-modified biochar (BS) had less carbon but more hydrogen and oxygen than biochar (B). The H/C and O/C ratios in sulfur-modified biochar were higher than those in biochar. The amount of CEC in sulfur-modified biochar was higher than that in biochar. The concentrations of K^+, Ca^{2+}, Mg^{2+}, and Na^+ cations in sulfur-modified biochar were lower than those in biochar. The water-holding capacity of biochar was 60% higher than that of sulfur-modified biochar (Table 1).

FTIR analysis of sunflower biochar (B and BS)

The sunflower biochar FTIR spectra result is shown in Fig. 3. Treatment B has a greater number of peaks than sulfur-modified biochar. Additionally, the peaks in BS are sharper than the B peaks. Peaks at 3419 cm^{-1} in the B treatment and 3402 cm^{-1} in BS correspond to the O-H hydroxyl groups of phenol (Goswami et al. 2016). The O-H hydroxyl group in BS is sharper than the B peak, which shows the high hydroxyl groups in BS. The peak at 2929 cm^{-1} in the biochar treatment represents C-H stretching that corresponds to methyl and methylene groups (Liu et al. 2018). The 1589 cm^{-1} peak in the BS

treatment and the 1576 cm^{-1} peak in the B treatment are related to the C=O bending of oxygen functional groups in carboxylic groups that was sharper in the BS treatment compared to the B treatment (Mahmoud et al. 2016). The peaks at 1433 cm^{-1} in the BS and B treatments represent C=C stretching, indicative of lignin and aromatic C that was sharper than in the B treatment compared to the BS treatment. The 1140 and 1192 cm^{-1} peaks, which are only observed in BS treatment, refer to the sulfur component, C=S stretching of thiocarbonyl groups (Burke and Fackler Jr 1972). Peaks at 1102 cm^{-1} in BS and 1106 cm^{-1} in B treatment correspond to the C-O stretching of secondary alcohols such as cyclohexane. The C-O peak in BS is sharper than the C-O peak in the B treatment. The 1033 cm^{-1} peak in the BS and B treatments represents the S=O bending of sulfoxide, which is very sharp in BS compared to the B treatment. The 875 cm^{-1} peaks in the BS and B treatments refer to C=C bending alkene vinylidene, which is sharper in the B treatment than in the BS treatment. The 780 and 718 cm^{-1} peaks in the B treatment and the 755 cm^{-1} peak in BS correspond to C-H stretching that is 1,2,3-trisubstituted and 1,2-disubstituted, respectively. The peak at 660 cm^{-1} in sulfur-enriched biochar refers to the C-S stretch of thioethers (Burke and Fackler Jr 1972). Peaks at 620 cm^{-1} in the B treatment and 602 cm^{-1} in the BS treatment represent S–S stretching, which is a disulfide component that was sharper in the BS treatment than in the B treatment. The 467 cm^{-1} peaks in BS and the 469 cm^{-1} peaks in the B treatment corresponded to the S–S stretch of aryl disulfides that was much sharper than in the BS treatment (Fig. 3).

Soil water content

Soil treated with 15 t ha^{-1} biochar (B) and sulfur-modified biochar (BS) increased the soil water content by 47% and 35%, respectively, at 20 days after irrigation compared to the control (without B and BS). Adding 15 t ha^{-1} biochar to soil enhanced the soil water content by 18% more than 15 t ha^{-1} sulfur-modified biochar ($p < 0.05$, Fig. 4).

Soil microbial and enzymatic activities

The application of 15 t ha^{-1} biochar (B) and sulfur–modified biochar (BS) enhanced MBC by 113% and 50%, respectively, compared to the control (without B and BS). Sulfur-modified biochar increased MBC by 63% less than biochar compared to the control treatment ($p < 0.05$,
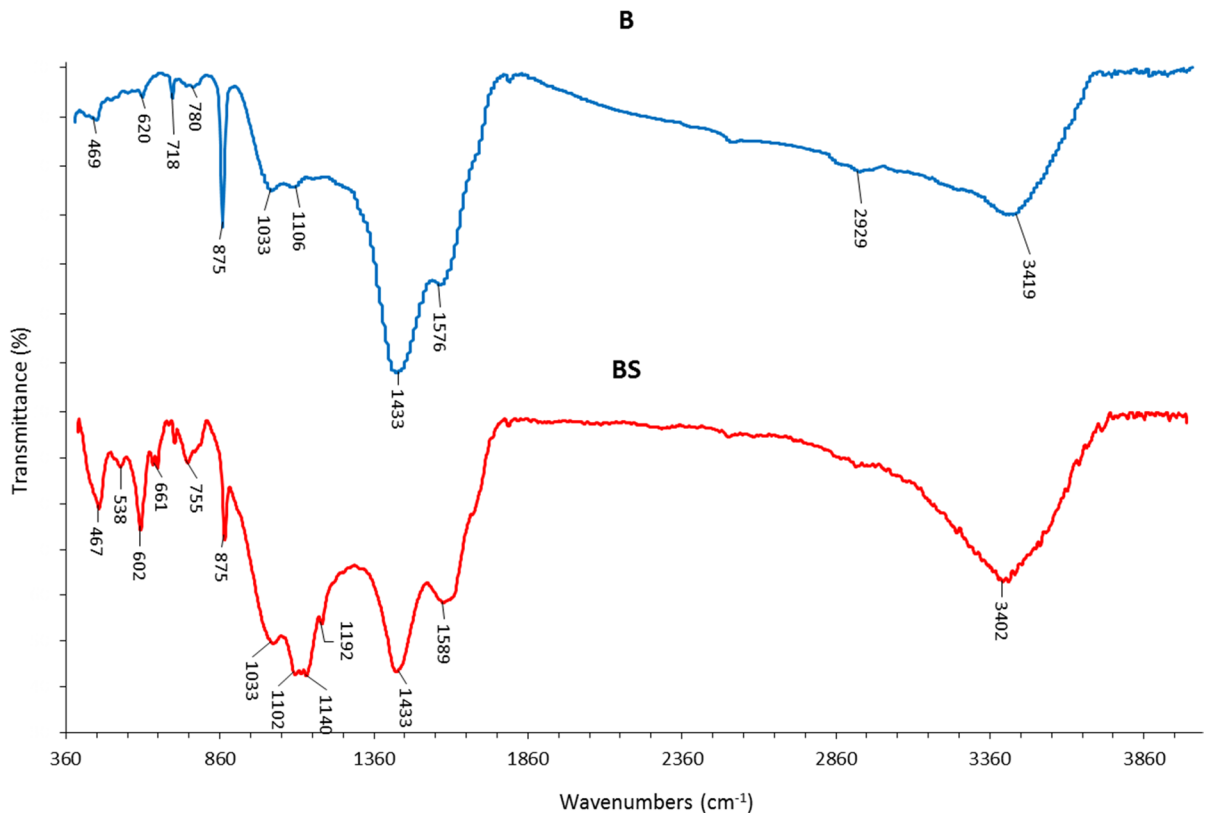
**Fig. 3** FT-IR spectra of biochar (B) and sulfur-modified biochar (BS)

Fig. 5). Soil BR in the presence of B and BS was 75% and 108% higher than that in the absence of biochar. Biochar and sulfur-modified biochar applications stimulated CAT activity by 13% and 30%, respectively, compared to the control. Soil treated with 15 t ha^{-1} biochar and sulfur-modified biochar increased ALP activity by 12% and 4%, respectively, compared to the control treatment. Sulfur-modified biochar increased ALP by 7% less than biochar compared to the control treatment ($p < 0.05$, Fig. 5). Soil amended with 15 t ha^{-1} B and BS promoted UA activity by 70% and 28% compared to the control, respectively. Sulfur-modified biochar increased UA by 40% less than biochar compared to the control treatment. Biochar and sulfur–modified biochar treatments increased DHA activity by 38% and 82% compared to the control treatment, respectively ($p < 0.05$, Fig. 5).

Soil chemical properties

Soil amended with 15 t ha^{-1} biochar (B) enhanced soil pH compared to the control (without B and BS), but soil amended with 15 t ha^{-1} sulfur-modified biochar (BS) decreased soil pH compared to the control ($p < 0.05$, Table 3). Soil treated with biochar and sulfur-modified biochar decreased soil EC by 17% and 5%, respectively, compared to the control treatment. The soil sodium adsorption ratio (SAR) in the presence of 15 t ha^{-1} biochar and sulfur-modified biochar was 14% and 7% lower than that in the control treatment, respectively. Biochar application increased DOC by 93%, and sulfur-modified biochar application enhanced DOC by 33% compared to the control. BS increased DOC by 60% less than B compared to the control treatment ($p < 0.05$, Table 3). Biochar and sulfur-modified biochar promoted AN by 74% and 48%, respectively, compared to the control treatment. Sulfur-modified biochar increased AN by 25% less than biochar compared to the control treatment. The application of B and BS treatments enhanced AP by 60% and 95% compared to the control, respectively ($p < 0.05$, Table 3).

Fig. 4 Soil water content. B: 15 t ha^{-1} biochar, BS: 15 t ha^{-1} sulfur-modified biochar
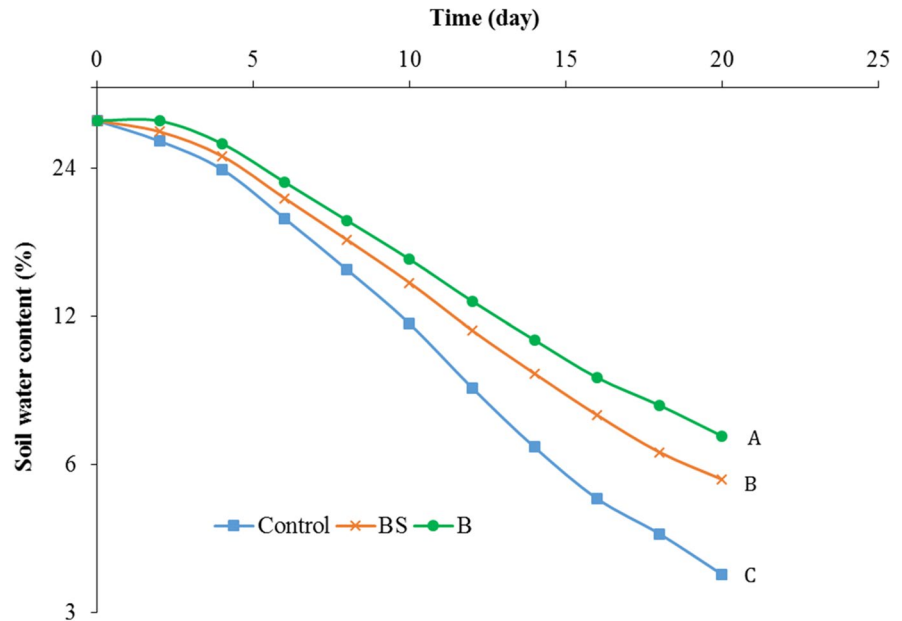
Millet plant properties

Experimental treatments improved plant nitrogen (N), phosphorus (P), and potassium (K). The application of biochar (B and BS) increased the plant nitrogen content by 7% and 11% compared to the control, respectively. Soil treated with B and BS treatments enhanced plant phosphorus content by 75% and 92% compared to the control treatment, respectively. Soil amended with B and BS treatments promoted plant potassium content by 56% and 35%, respectively, compared to the control ($p<0.05$, Table 4). Soil treated with biochar and sulfur-modified biochar improved RWC by 40% and 13%, respectively, compared to the control treatment. Biochar (B) increased RWC by 27% more than BS compared to the control treatment. Application of B and BS treatments increased plant yield by 58% and 115%, respectively, compared to the control ($p<0.05$, Table 4).

Correlation between soil and plant parameters

The Pearson correlation coefficient between soil and plant factors is shown in Fig. 6. Soil EC and SAR had a negative and significant correlation ($p<0.01$) with most of the measured parameters, especially with soil microbial and enzymatic activity (MBC, ALP, and UA) and plant properties (yield, RWC, and plant nutrients). Soil pH had a positive correlation with RWC and DOC and a negative correlation with ALP and AP ($p<0.01$, Fig. 6). There was a positive correlation ($p<0.01$) between DOC

and MBC, ALP, UA, and AN. Soil available nitrogen (AN) had a positive correlation with MBC, UA, and RWC ($p<0.01$, Fig. 6). AP had the highest positive correlation ($p<0.01$) with BR, DHA, CAT, and plant yield. There was a positive correlation between UA activity, MBC, and RWC ($p<0.01$). ALP activity had a positive correlation with MBC. CAT and DHA activity had the highest positive correlation with plant yield and BR ($p<0.01$, Fig. 6). MBC had a positive correlation with RWC. There were positive correlations between plant yield and AP, CAT, DHA, BR, plant N, and plant P ($p<0.01$).

Relative importance index

The results show that BR, AP, DHA, and CAT had the greatest impact on millet yield, while EC, pH, and AN had secondary relative importance for the millet plant. According to the results, biological parameters (CAT, DHA, and BR) had a greater impact on millet yield than chemical parameters (Fig. 7).

Discussion

Biochar characteristics

The presence of sulfate ions in sulfur-modified biochar increased CEC compared to biochar. This change in CEC can be attributed to the alteration in the number of functional groups in biochar after modification with
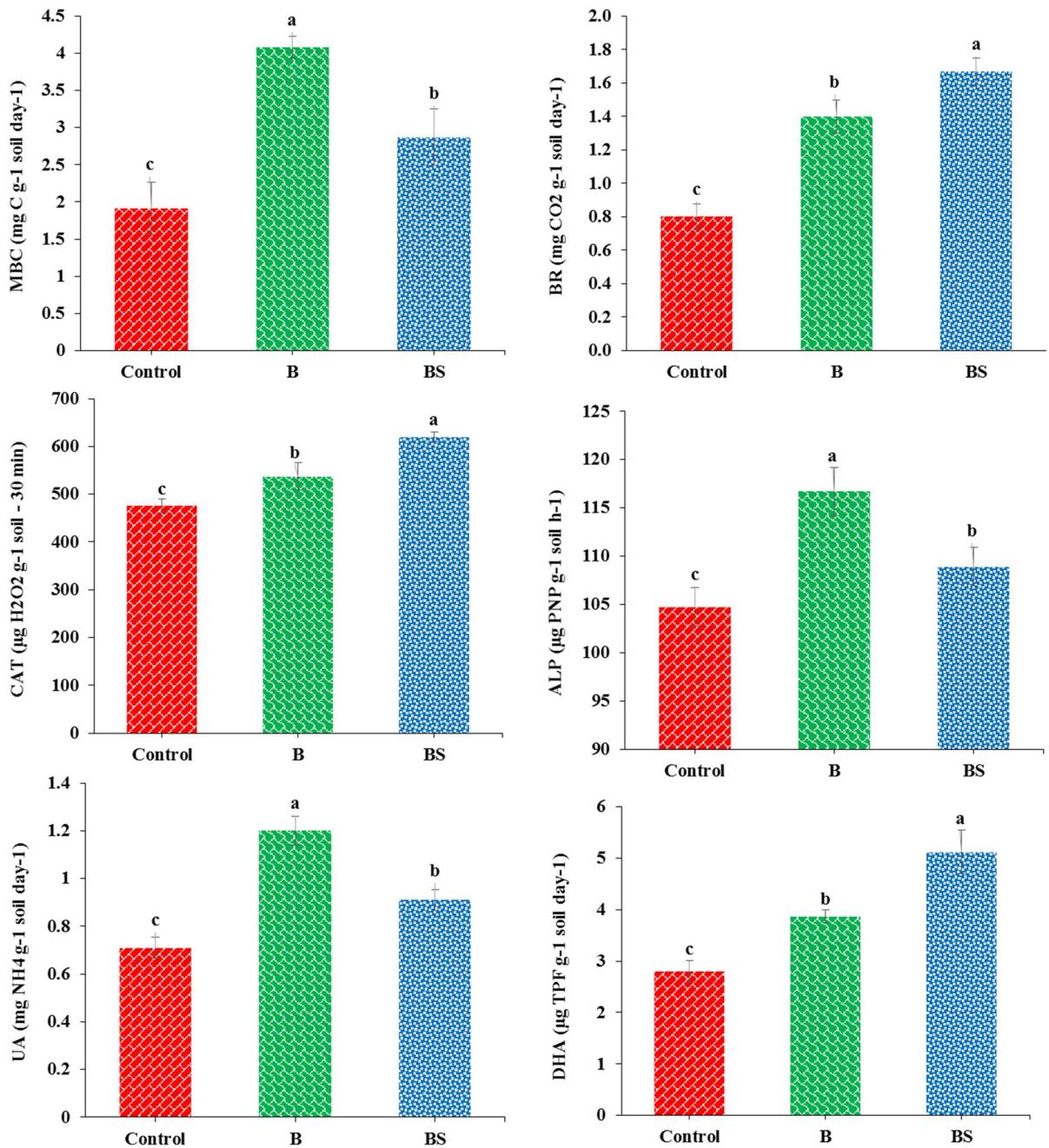
Fig. 5 Soil microbial and enzymatic activities. B: 15 t ha^{-1} biochar, BS: 15 t ha^{-1} sulfur-modified biochar. CAT: catalase activity, ALP: alkaline phosphatase activity, UA: urease activity, DHA: dehydrogenase activity, MBC: microbial biomass carbon, BR: basal respiration. Different letters show significant differences among treatments at $p < 0.05$. Error bars indicate standard deviation ($n = 3$)

sulfur (Fig. 3). The enhancement of hydroxyl and carboxyl functional groups in biochar increases the negative surface charge and boosts CEC (Janu et al. 2021). The modification of biochar with sulfur has increased

functional groups (Fig. 3) and enhanced active sites on the surface of biochar, which can improve the ion absorption power of BS treatment (Nguyen et al. 2019). According to the results, the concentration

Table 3 The effect of treatments on soil chemical properties

Treatments	pH	ECe	SAR	DOC	AN	AP
	-	(dS m^{-1})	-	(mg kg^{-1})		
Control	7.96 ± 0.03b	9.66 ± 0.2a	16.83 ± 0.3a	8.12 ± 0.7c	13.88 ± 2c	3.06 ± 0.5c
B	8.26 ± 0.06a	7.95 ± 0.1c	14.5 ± 0.1c	15.70 ± 0.5a	24.16 ± 0.9a	4.92 ± 0.9b
BS	7.83 ± 0.04c	9.20 ± 0.08b	15.7 ± 0.15b	10.82 ± 0.3b	20.60 ± 1b	6.01 ± 0.3a

B: 15 t ha^{-1} biochar, *BS*: 15 t ha^{-1} sulfur-modified biochar. *DOC*: dissolved organic carbon, *AN*: available nitrogen, *AP*: available phosphorus. Different letters show significant differences among treatments at $p < 0.05$. Error bars indicate standard deviation ($n = 3$)

Table 4 The effect of treatments on plant properties

Treatments	N	P	K	RWC	Yield
	%	g kg^{-1}	%	%	(t ha^{-1})
Control	2.37 ± 0.04c	1.11 ± 0.2c	1.50 ± 0.09c	81 ± 4c	1.53 ± 0.1c
B	2.53 ± 0.06b	1.96 ± 0.05b	2.34 ± 0.06a	113 ± 7a	2.43 ± 0.3b
BS	2.64 ± 0.03a	2.14 ± 0.02a	2.07 ± 0.04b	92 ± 2b	3.30 ± 0.07a

B: 15 t ha^{-1} biochar, *BS*: 15 t ha^{-1} sulfur-modified biochar. *N*: nitrogen content, *P*: phosphorus content, *K*: potassium content. *RWC*: relative water content. Yield: Total dry weight plant. Different letters show significant differences among treatments at $p < 0.05$. Error bars indicate standard deviation ($n = 3$)

of H in BS was higher than that in the B treatment (Table 1). The higher molar ratio of O:C and H:C in sulfur-modified biochar compared to biochar reduces carbon stability and lowers the aromatic carbon content so that its organic compounds are more easily decomposed (Domingues et al. 2017). The water-holding capacity of BS was higher than that of the B treatment (Table 1). According to Fig. 1, the drainage water from the water-holding capacity test of biochar is black, but the water from sulfur-modified biochar is yellow. The black color of the biochar extract can be due to carbon compounds such as humic acid. It is possible that during the BS treatment, sulfur reacted with carbon, resulting in carbon deposition. The inactivation of carbon in the pore wall disrupts the empty pore structure and reduces its water-holding capacity compared to biochar. Liu et al. (2020) reported that biochar modified with H_2SO_4 decreases the specific surface area and total pore volume but increases the average pore size. The low acidity of biochar modified with sulfuric acid weakened the structure of biochar and blocked its pores. In the BS treatment, C=C stretching at the peak of 1433 cm^{-1}, indicative of lignin and aromatic C, shifted to C=S stretching at the peaks of 1140 and 1192 cm^{-1}, corresponding to increased carbon-sulfur components such as thiocarbonyl groups, according to FTIR results (Fig. 3).

Soil enzymatic activity (DHA, ALP, CAT and UA)

Sulfur-modified biochar increased DHA more than biochar (Fig. 5). In the biochar component, the higher the molar ratio of O/C and H/C, the lower the carbon stability and aromatic carbon content, so that the organic compounds are easily decomposed and increase the enzymatic activity of the soil (Domingues et al. 2017). The molar ratio of O/C and H/C in BS is higher than in the B treatment (Table 1). The correlation results (Fig. 6) between the data showed that dehydrogenase activity had a positive correlation with soil microbial respiration (BR) and available phosphorus (AP). Biochar provides available substrate (carbon and nutrients) to the microbial population and stimulates soil enzymatic activity (Palansooriya et al. 2019). In general, the results of this study showed the positive effect of biochar and sulfur-modified biochar on increasing the activity of the dehydrogenase enzyme as an intracellular enzyme effective in the cellular metabolism of soil microorganisms and cell protection against reactive oxygen species such as hydrogen peroxide. According to the results, B and BS stimulated ALP activity (Fig. 5). The application of sulfur-modified biochar reduced the increasing trend of alkaline phosphatase activity compared to biochar treatment. Sulfur-modified
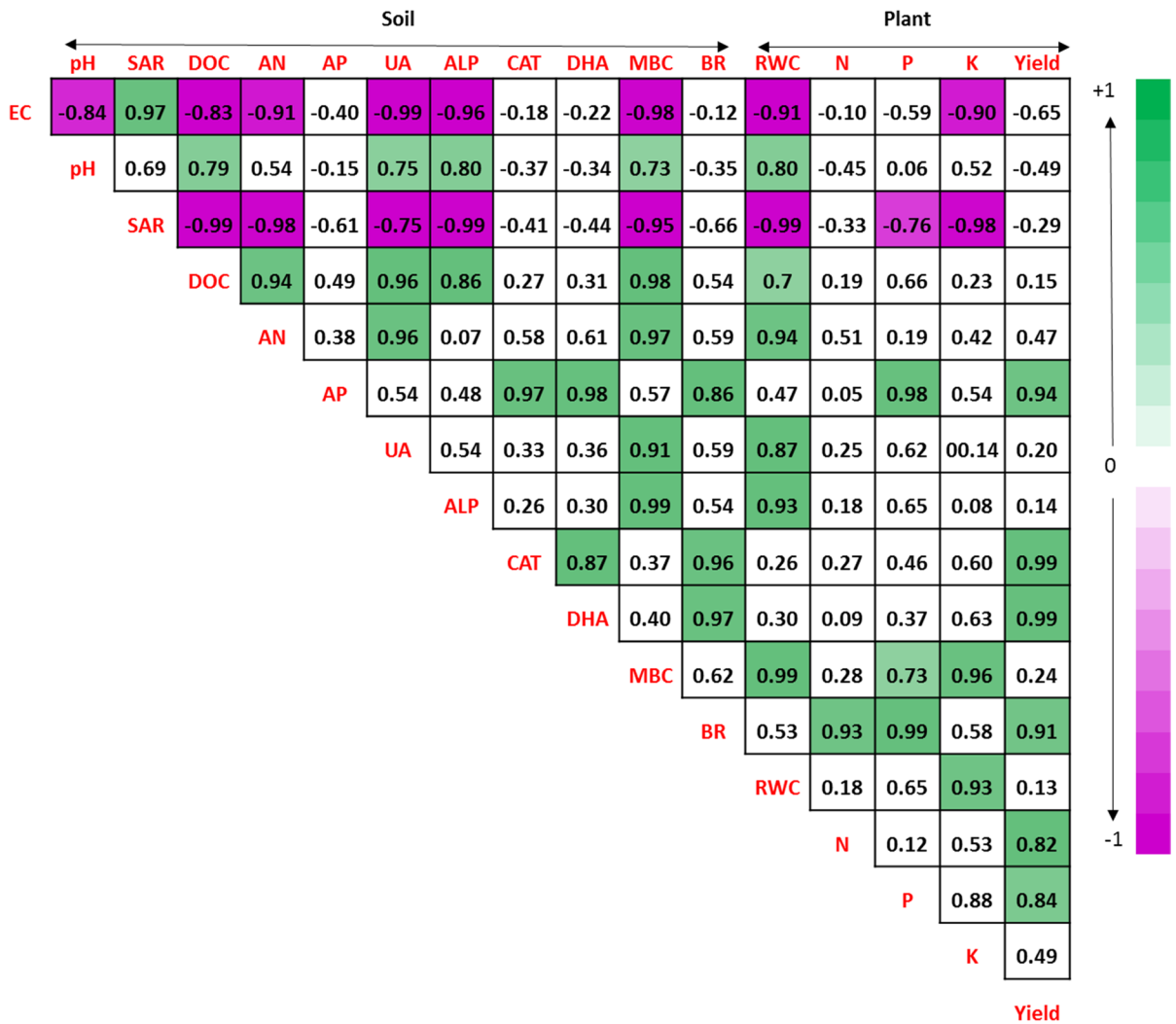
	Soil											Plant				
	pH	SAR	DOC	AN	AP	UA	ALP	CAT	DHA	MBC	BR	RWC	N	P	K	Yield
EC	-0.84	0.97	-0.83	-0.91	-0.40	-0.99	-0.96	-0.18	-0.22	-0.98	-0.12	-0.91	-0.10	-0.59	-0.90	-0.65
pH		0.69	0.79	0.54	-0.15	0.75	0.80	-0.37	-0.34	0.73	-0.35	0.80	-0.45	0.06	0.52	-0.49
SAR			-0.99	-0.98	-0.61	-0.75	-0.99	-0.41	-0.44	-0.95	-0.66	-0.99	-0.33	-0.76	-0.98	-0.29
DOC				0.94	0.49	0.96	0.86	0.27	0.31	0.98	0.54	0.7	0.19	0.66	0.23	0.15
AN					0.38	0.96	0.07	0.58	0.61	0.97	0.59	0.94	0.51	0.19	0.42	0.47
AP						0.54	0.48	0.97	0.98	0.57	0.86	0.47	0.05	0.98	0.54	0.94
UA							0.54	0.33	0.36	0.91	0.59	0.87	0.25	0.62	00.14	0.20
ALP								0.26	0.30	0.99	0.54	0.93	0.18	0.65	0.08	0.14
CAT									0.87	0.37	0.96	0.26	0.27	0.46	0.60	0.99
DHA										0.40	0.97	0.30	0.09	0.37	0.63	0.99
MBC											0.62	0.99	0.28	0.73	0.96	0.24
BR												0.53	0.93	0.99	0.58	0.91
RWC													0.18	0.65	0.93	0.13
N														0.12	0.53	0.82
P															0.88	0.84
K																0.49
Yield																

Fig. 6 Pearson correlation coefficients between soil and plant properties. MBC: microbial biomass carbon, YIELD: millet yield, DOC: dissolved organic carbon, CAT: catalase activity, ALP: alkaline phosphatase activity, AN: available nitrogen, UA: urease activity, AP: available phosphorus, BR: basal respiration, DHA: dehydrogenase activity. RWC: relative water content. N, P, and K: nitrogen, phosphorus, and potassium in the plant

biochar reduced soil pH and subsequently increased soil phosphorus solubility (Table 3). Increasing phosphorus in the soil solution will reduce alkaline phosphatase activity (Khadem and Raiesi 2017). Additionally, the results of the correlation between the data showed a positive correlation between alkaline phosphatase activity and soil pH (Fig. 6). Ghoularata et al. (2008) indicated that the available phosphorus in the soil solution is a controlling factor in the synthesis and release of phosphatases by microorganisms and plants, which reduces the enzymatic content of phosphatases in the soil. The use of sulfur reduces ALP due to lower soil pH (Gupta and Germida 1988). The results show that sulfur-modified biochar reduced the increasing trend of urease activity; sulfur most likely acts as an inhibitor of urease activity. Gupta and Germida (1988) reported that the application of sulfur to soil reduced the activity of the enzyme urease due to the reduction in the population of protozoans and the reduction in the nitrification process. Baligar et al. (2005) reported that there is a positive relationship between urease activity and soil organic carbon.
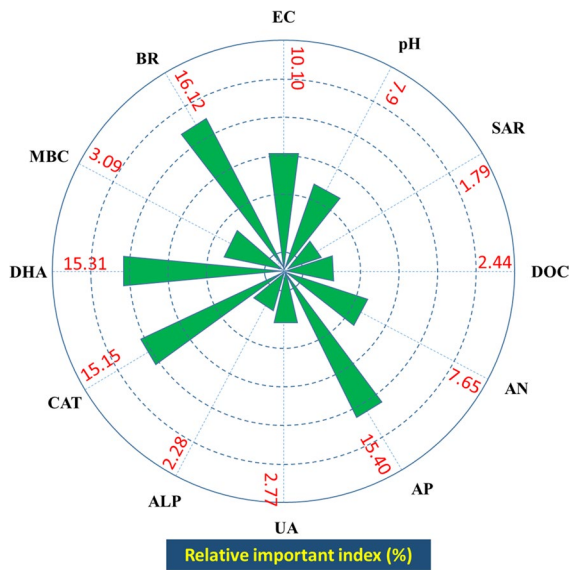
Fig. 7 Relative importance index between soil properties and millet yield. MBC: microbial biomass carbon, DOC: dissolved organic carbon, CAT: catalase activity, ALP: alkaline phosphatase activity, AN: available nitrogen, UA: urease activity, AP: available phosphorus, BR: basal respiration, DHA: dehydrogenase activity

The results show that biochar treatments (B and BS) increased catalase activity (Fig. 5). Sulfur-modified biochar treatment increased catalase activity more than biochar. Zhao et al. (2008) reported that long-term soil treatment with sulfur fertilizers increased catalase activity. In general, catalase activity is higher in high-quality soils but decreases in soils with a high pH or temperature (Wang et al. 2016). Accordingly, since biochar treatment increased soil pH (Table 3), it had less catalase activity than biochar modified with sulfur.

Microbial activity (MBC and BR)

According to the results, biochar and sulfur-modified biochar increased MBC (Fig. 5). The increase in MBC indicates the growth of soil microorganisms, which could be due to increased carbon and nutrients. Due to its porous structure, biochar creates a suitable environment for the growth of soil microorganisms, especially bacteria (Zhu et al. 2017). Sulfur-modified biochar, in contrast to biochar, reduced the increasing trend of MBC (Fig. 5); this result may be due to the reduction in dissolved carbon in soil (Table 3). The

use of sulfur reduces the pH of the soil; acidification of the soil increases the solubility of soil organic matter and causes leaching of dissolved carbon from biologically active points (Stroo and Alexander 1986). Correlation analysis also showed a positive relationship between MBC, soil pH, and DOC (Fig. 6). The porous structure of biochar, its high specific surface area, and its ability to absorb soluble organic matter, gases, and minerals provide a suitable place for the habitat and growth of soil microorganisms (Lehmann et al. 2011). In biochar pores, water and a variety of gases, including carbon dioxide and oxygen, are either dissolved in solution or adsorbed to the biochar surface (Shinogi and Kanri 2003). Application of B and BS treatments enhanced BR (Fig. 5). Soil microbial respiration depends on soil porosity, gas concentration, diffusion rate, and aerobic or anaerobic conditions (Lehmann et al. 2011). Biochar compounds have the ability to absorb volatile substances on their surface and slowly provide them as an accessible substrate to soil microorganisms, increasing the population and soil microbial respiration (Rutigliano et al. 2014). The amount of volatiles in sulfur-modified biochar was higher than that in biochar (Table 1).

Soil water content

Sunflower biochar (B and BS) enhanced the soil water content; BS had a lower effect on the soil water content than the B treatment (Fig. 4). The biochar used in this study (B and BS) have a high water-holding capacity (400% and 250%) compared to other biochar compounds; for example, the water-holding capacity of coconut shell biochar is 60% (Pituya et al. 2017; Paul and Harikumar 2022). This WHC of sunflower biochar is due to the superabsorbent materials of the sunflower stalk (pith fiber and hemicellulose). The water-holding capacity of the pith fiber of a sunflower stalk is 40 g of water per g of fiber (Qi 2017). Modification of biochar with sulfur decreased the WHC of BS and soil water content compared to the B treatment. Sulfur probably destroyed the internal spaces of the biochar by loosening the carbon bonds of the biochar and reducing the water-holding capacity of sulfur-modified biochar. Liu et al. (2020) reported that biochar modified with H_2SO_4 decreases the specific surface area and total pore volume; the low acidity of biochar modified with sulfuric acid weakened the structure of biochar and blocked its pores. Peak

intensities of C=C stretching (1576 and 1433 cm^{-1}) are higher in the B treatment than in the BS treatment (Fig. 3). Functional groups of C=C that indicated lignin in BS shifted to the C=S stretching of the sulfur component (1140 and 1192 cm^{-1}).

Soil properties

Biochar (B) increased soil pH, but sulfur-modified biochar decreased soil pH (Table 3). Additionally, the pH of sulfur-modified biochar was lower than that of biochar (Table 1). The increase in sulfur oxidation caused an increase in the concentration of sulfate ions and a decrease in the pH of biochar. There is a negative correlation between sulfate ions and soil pH (Maynard et al. 1986). Biochar is a moisture-absorbing compound that is porous and has a high specific surface area, so combining sulfur with biochar will provide a suitable environment for the activity of sulfur-oxidizing microorganisms and reduce the pH of biochar and soil. Grafe et al. (2021) reported that sulfur-modified biochar reduced the pH of soil. Biochar and sulfur-modified biochar treatments decreased soil EC and SAR; biochar decreased soil EC and SAR more than sulfur-modified biochar. Sulfur-modified biochar had a higher water-holding capacity than biochar (Table 1). There is an enhanced separation of salts such as Na+ from the surface of soil colloids and leaching salt from the root area; in addition, the CEC of sulfur-modified biochar is higher than that of biochar (Table 1). Because it can absorb some harmful ions, such as sodium, it reduces soil salinity (Yang et al. 2020). The correlation results in Fig. 5 show a negative correlation of soil EC and SAR with all measured parameters, especially soil biological parameters. Biochar increased soil-dissolved organic carbon (DOC), but sulfur-modified biochar reduced the increasing trend of DOC (Table 3). Sulfur-modified biochar has increased sulfate ions and decreased pH in the soil. Soil acidification increases the solubility of carbon and increases the leaching of dissolved carbon (Stroo and Alexander 1986). The presence of sulfur in the soil solution can be complex with dissolved carbon and cause carbon deposition (Koch et al. 2017). There was a positive correlation between DOC and the pH of the soil (Fig. 6). Biochar and sulfur-modified biochar treatments increased the amount of available phosphorus in the soil (Table 3). Sulfur-modified biochar increased phosphorus availability more than biochar. Sulfur-modified biochar

decreased soil pH (Table 3) and thus improved soil phosphorus availability. Biochar and sulfur-modified biochar treatments increased the available nitrogen in the soil. BS was lower than that of B treatment-enhanced AN (Table 3). Biochar increased the urease enzyme by providing suitable conditions for the growth of soil microorganisms (Fig. 5), followed by an enhancement in the concentration of ammonium and nitrate in the soil solution. Although the total nitrogen in sulfur-modified biochar was higher than that in biochar (Table 1), the available nitrogen (AN) in the soil treated with BS was lower than that in the B treatment. Sulfur probably has an inhibitory effect on the enzyme urease (Fig. 5) and reduces the availability of nitrogen concentrations in the soil (Crusciol et al. 2019).

Plant parameters

Based on the results, biochar (B, BS) treatments increased the yield of millet plants (Table 4). Biochar improves the physical and chemical conditions of the soil, such as soil water content (Fig. 4) and nutrient solubility (phosphorus, for example), and creates an environment conducive to the growth of beneficial soil microorganisms and subsequent plant yield development. Based on the correlation results, biological parameters had the highest effect on increasing millet yield (Fig. 6). The results show that RWC increased in the presence of B and BS treatments, and BS enhanced RWC at a lower level than B treatment. There is a positive correlation between RWC and soil water content (Huang et al. 2020). Biochar increased the RWC in the plant by increasing the soil water content (Fig. 4). According to the results of the relative importance index (Fig. 7), biological parameters such as DHA, CAT, and soil-available phosphors had a high effect on plant yield. Since the soil in this study is calcareous and deficient in phosphorus (Table 2), sunflower biochar, especially sulfur-modified biochar, decreases soil pH, and its phosphorus content enhances plant phosphorus and promotes plant yield (Table 4). In saline and sodic soils with high salt concentrations, the presence of toxic ions such as sodium and the accumulation of reactive oxygen species (ROS) in microbial biomass reduce the microbial and enzymatic activities of the soil (Kumawat et al. 2022). Strengthening oxidoreductase activities such as catalase and dehydrogenase can protect the microbial community from ROS compounds, and

improving the activity of other enzymes such as urease and phosphatase can help to provide nutrients and plant growth (Zandi and Schnug 2022). There were positive correlations between CAT and DHA and soil AP and AN (Fig. 6).

Conclusion

This study showed that the application of sunflower biochar (B and BS) promoted soil water content by 47% and 35%, respectively, compared to the control. B and BS treatments by reducing soil EC and SAR stimulated soil microbial and enzymatic activity, which enhanced the availability of nutrients (AN and AP) and millet yield. Modification of biochar with sulfur decreased the water-holding capacity of BS by 60% but also decreased the pH of BS by 3 units compared to the B treatment, and by providing more nutrients to the plant, enhanced plant yield by 57% more than the simple biochar treatment (B). This study demonstrates that using sunflower biochar (B and BS) in saline-sodic and calcareous soil increased millet yield by improving soil water content and soil nutrients (especially phosphorus), decreasing soil salinity, and stimulating soil microbial and enzymatic activity (especially DHA and CAT).

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